











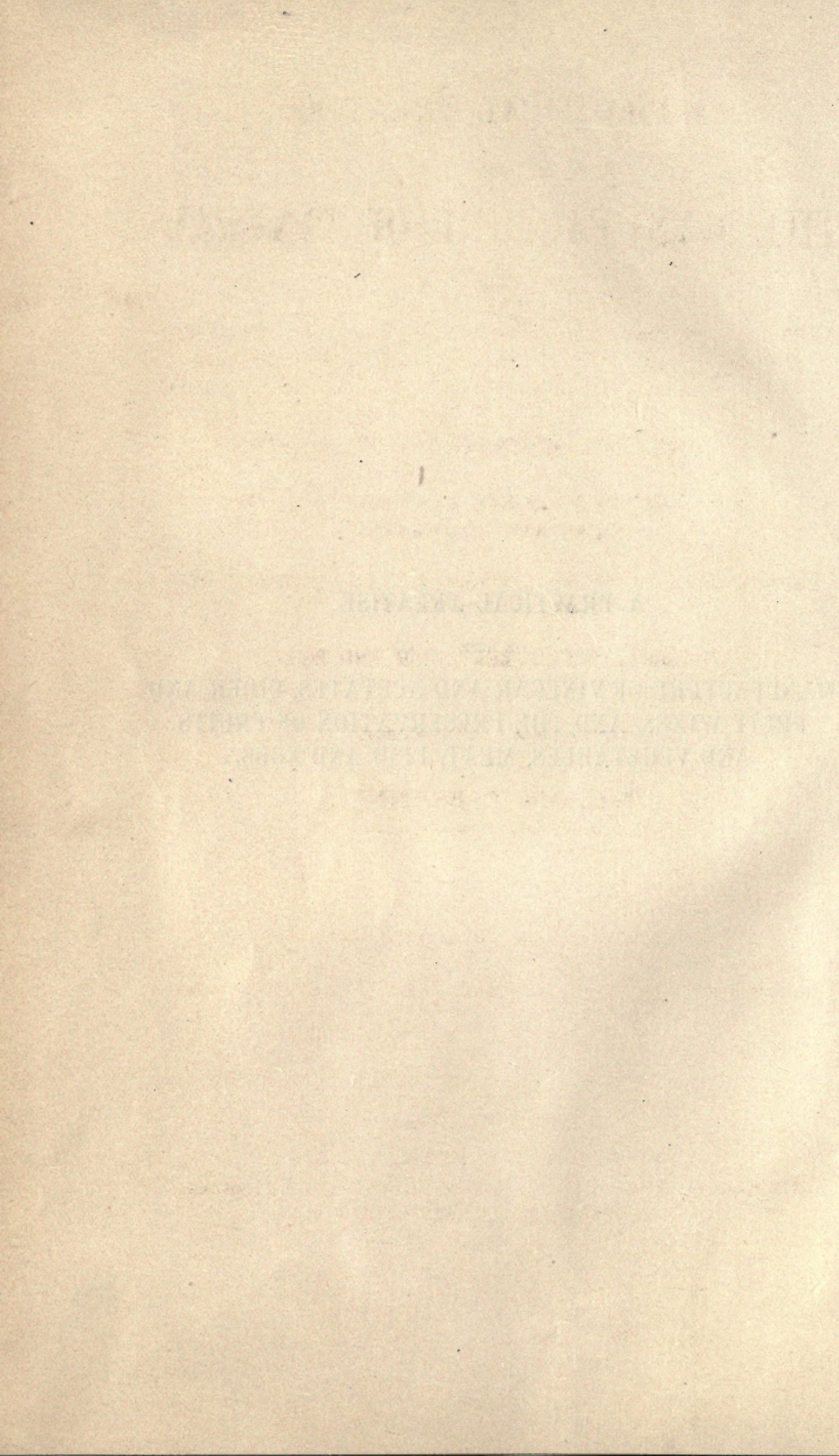




A PRACTICAL TREATISE

ON THE

MANUFACTURE OF VINEGAR AND ACETATES, CIDER AND  
FRUIT WINES, AND THE PRESERVATION OF FRUITS  
AND VEGETABLES, MEAT, FISH AND EGGS.





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**A PRACTICAL TREATISE**  
**ON**  
**THE MANUFACTURE OF VINEGAR,**

**WITH**

**SPECIAL CONSIDERATION OF WOOD VINEGAR AND OTHER BY-  
PRODUCTS OBTAINED IN THE DESTRUCTIVE DISTILLATION  
OF WOOD; THE PREPARATION OF ACETATES.**

**MANUFACTURE OF CIDER AND FRUIT-WINES;**

**PRESERVATION OF FRUITS AND VEGETABLES BY  
CANNING AND EVAPORATION;**

**PREPARATION OF FRUIT-BUTTERS, JELLIES, MARMALADES,  
PICKLES, MUSTARDS, ETC.**

**PRESERVATION OF MEAT, FISH AND EGGS.**

**EDITED FROM VARIOUS SOURCES**

**BY**

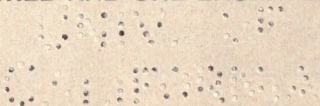
**WILLIAM T. BRANNT,**

**EDITOR OF "THE TECHNO-CHEMICAL RECEIPT BOOK."**

**THIRD EDITION.**

**THOROUGHLY REVISED AND LARGELY RE-WRITTEN.**

**ILLUSTRATED BY ONE HUNDRED AND ONE ENGRAVINGS.**



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## PREFACE TO THE THIRD EDITION.

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THE second edition of A PRACTICAL TREATISE ON THE MANUFACTURE OF VINEGAR has been out of print for sometime, and there being no recent work to take its place notwithstanding the constant demand for a book on the various important branches of industry treated of, the publication of a new edition was deemed advisable.

Like the previous editions the volume is divided into three parts.

Part I. is devoted to the Manufacture of Vinegar, and includes the production of wood vinegar and other by-products obtained in the destructive distillation of wood, as well as the preparation of acetates.

Part II. contains the Manufacture of Cider, Fruit Wines, etc., and Part III. the Preservation by various methods of Fruit and Vegetables, and of Meat, Fish and Eggs.

In this, the third edition, no essential changes have been made in the arrangement of the book, but it has been thoroughly revised and largely re-written, old and obsolete matter having been eliminated and new material introduced. The best authorities have been diligently consulted and freely drawn on, for which due credit has, whenever possible, been given in the text.

It is hoped that this new edition will meet with the same favorable reception as the previous ones, and that it will be of practical utility.

A copious table of contents as well as a very full index will render reference to any subject in the book prompt and easy.

PHILADELPHIA, November 16, 1914.

W. T. B.





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# PART I.

## VINEGAR.

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### CHAPTER I.

#### INTRODUCTORY AND HISTORICAL.

Ordinary vinegar consists of a weak solution of acetic acid in aqueous fluids prepared by the oxidation of alcoholic liquors by means of acetic acid bacteria, *Bacterium aceti*, of which there are many varieties, the best races being propagated by pure culture methods, and used for impregnating the alcoholic liquors to be fermented. The color of vinegar and, to a certain extent, also its odor and taste are influenced by the materials from which it is prepared. In this form it has been known from the earliest times, and it must have been used contemporaneously with wine, because it is evident that at the temperature of the Eastern countries, where the first experiments with the juice of the grape were made, fermentation must have set in rapidly and the wine been quickly transformed into an acid compound. Vinegar is mentioned in the Old Testament, and Hippocrâtes made use of it as a medicine. That the solvent effects of vinegar were understood by the ancients, is shown by the well-known anecdote of Cleopatra, related by Pliny. To gain a wager that she could consume at a single meal the value of a million sesterces, she dissolved pearls in vinegar which she drank. This is also shown by the equally well known, but exaggerated account by Livy and Plutarch, that Hannibal overcame the difficulties offered by

the rocks to the passage of his army over the Alps, by dissolving them with vinegar. Admitting the exaggeration, or the explanation which some give, viz.: that Hannibal used the vinegar by way of strategem, to incite his men to greater exertion by the belief that the difficulties of the path were diminished, the case nevertheless shows that the solvent action of vinegar upon certain substances was well known at that period.

Vitruvius also states that rocks which cannot be attacked by either fire or iron, will yield when heated and wet with vinegar.

Although there can be no doubt that vinegar was in very general use at an early period, there was no definite knowledge as to the cause of its production and the mode of its formation, and we are indebted to the much-abused alchemists for the first knowledge of its purification and concentration by distillation.

Gerber, who flourished in the eighth century, gives us the earliest description of the process of increasing the strength of wine-vinegar by distillation, and Albucases, about 1100, stated the fact that vinegar to be colorless has to be distilled over a moderate fire. Basilius Valentinus, a monk and celebrated alchemist of the fifteenth century, knew that by the slow distillation of vinegar, first a weak, and then a stronger product is obtained, and he was probably also acquainted with the process of obtaining strong acetic acid by distilling cupric acetate (*verdigris*.) In fact, for a long time this was the only way of preparing acetic acid, the result of the further rectification of the product being termed *radical vinegar*, *spiritus Veneris*, *Venus's vinegar*, *spiritus aeruginis*, etc.

Stael, in 1697, strengthened vinegar by freezing out some of its water. In 1702, he taught the method of obtaining strong acetic acid by neutralizing vinegar by an alkali, and distilling the acetate thus formed with oil of vitriol. The Count de Laragnais (1759), and the Marquis de Courtenvaux (1768), showed that the most concentrated acetic acid obtained from

verdigris was capable of crystallization. Loewitz (1789) taught how pure, but weak acetic acid might be strengthened by passing it repeatedly over charcoal powder. It may thus be deprived of so much of its water that it crystallizes by cold. This crystallizable acetic acid is the strongest which it is possible to obtain. Durande (1777), gave to it the name which it still bears, of *glacial acetic acid*.

The formation of an acid body in the destructive distillation of wood was known as early as the seventeenth century. However, it was for a long time not recognized as acetic acid, but considered a special acid (pyroligneous acid). Fourcroy and Vauquelin, in 1800, were the first to recognize this acid as acetic acid, and Thénard, in 1802, demonstrated the presence of acetic acid among the products formed in the destructive distillation of animal substances.

Berzelius, in 1814, determined the exact chemical constitution of acetic acid, and Saussure, in the same year, that of alcohol. Dr. J. Davy observed that spongy platinum, in contact with vapor of alcohol, became incandescent and generated acetic acid. Döbereiner further studied the nature of the acid, and proved that the alcohol was oxidized at the expense of the atmospheric air, producing acetic acid and water, and that no carbonic acid was formed, thus pointing out the fallacy of the opinion held by the chemists of his time that carbonic acid was one of the products of acetic fermentation.

Schützenbach, in 1823, one year after the establishment by Döbereiner of the now generally accepted theory of the formation of acetic acid from alcohol, introduced the quick process of manufacturing vinegar.

Without detracting from the credit due to Schützenbach for the introduction of his method and the improvement in the process of manufacturing vinegar, it may be mentioned that as early as 1732, nearly a century before, the celebrated Dutch chemist and physician, Boerhave, made known a method for making vinegar from wine, which contained the principles of the quick process.



Although it is now more than ninety years since the introduction of Schützenbach's process into practice, the manufacture of vinegar from alcohol remains nearly the same. While no change can be made as regards the theoretical part of the process, it being erected on a foundation clearly indicated by a knowledge of natural laws, many important improvements may surely be introduced in the manufacture of vinegar on a large scale, this being especially the case where it is uninterruptedly carried on with the use of suitable apparatus. Many manufacturers still work according to Schützenbach's original plan, *i. e.*, they use an immense amount of labor for a performance which can be attained in a much simpler manner.

Progress is essential in every business, but for several reasons it is especially necessary for the manufacturer engaged in making vinegar by the quick process. Alcohol in every form—whiskey, beer, wine—is everywhere subjected to a high tax, and the constantly increasing taxation of this fundamental material for the manufacture of vinegar, of course increases the price the manufacturer has to pay for it. Another reason why the production of vinegar from alcohol becomes constantly more difficult is found in the great competition arising from the continued improvements in the manufacture of pure acetic acid from wood. Not many years ago it was considered impossible to obtain entirely pure acetic acid from wood when manufacturing on a large scale, but the article produced at the present time may be almost designated as "chemically pure" in the true sense of the word, it containing, besides acetic acid, only water, and the most accurate analysis cannot detect a trace of the products of tar, which render unpurified wood vinegar unfit for use.

For consumption on a large scale, especially where only a body of an acid taste is required, the use of so-called "vinegar essence," *i. e.*, pure 80 to 90 per cent. acetic acid, obtained from wood, and which, when properly diluted, furnishes ordinary vinegar, will undoubtedly gradually supersede vinegar prepared from alcohol, it being considerably cheaper. And not-

withstanding that the price of wood vinegar is declining every year, in regions where wood is plentiful and cheap its manufacture is a remunerative industry on account of the many valuable by-products—tar, wood spirit, charcoal—obtained besides acetic acid. At the present time, for all industrial purposes where acetic acid is required, as for instance in the manufacture of tar colors, that obtained from wood is used, and the quantities consumed in the production of vinegar for domestic purposes becomes larger every year.

But the manufacture of vinegar from alcohol and alcoholic liquids will nevertheless continue to flourish, because the product obtained from them possesses different properties from the pure acetic acid prepared from wood. Vinegar obtained from alcohol, and still more so that from fermented fruit juices, such as wine, cider, skins of pressed grapes, or from malt, contains, besides acetic acid and water, small quantities of bodies which, on account of their being analogous to those occurring in wine, may be designated as “bouquet bodies,” and which give to the vinegar an agreeable smell and taste entirely wanting in acetic acid prepared from wood. These properties are so characteristic that any one gifted with a sensitive and practiced sense of smell can at once distinguish pure acetic acid vinegar from that prepared from wine, cider, beer, etc.

By the addition of volatile oils or compound ethers an agreeable odor may, of course, be imparted to vinegar obtained by diluting pure wood acetic acid with water, but it is impossible to produce the harmonious bouquet peculiar to vinegar prepared from alcohol or fruit juices, a similar relation existing here as between genuine and artificial wine. The latter may be made so that, as regards taste and smell, it nearly approaches genuine wine, but a connoisseur will at once detect the difference.

The principal defects of the manner of manufacturing vinegar by the quick process in general use are not in the method itself, for that, as already indicated, corresponds entirely to the theoretical conditions, and yields as good a product as can be

obtained from the raw material used. The weak point of the process is found in practical execution of it, the losses of material being much more considerable and greater than absolutely necessary ; the consumption of labor is large, and, as every manufacturer knows from experience, interruptions in the regular process of working are of too frequent occurrence.

All these disadvantages can be reduced to a minimum, if not absolutely overcome, and it is hoped sufficient hints how this can be done will be found in the following chapters.

As will be explained later on acetic acid contains the same elements found in carbonic acid and water, and to judge from the results already attained by chemistry in building up compounds from their elements, a method will no doubt be found by which acetic acid can on a large scale be produced from its elements. It is difficult to predict the effect the discovery of such a process would have upon the life of all other methods of vinegar manufacture. In fact, acetic acid has already been prepared in this manner, but the method employed is not adapted for operations on a large scale.

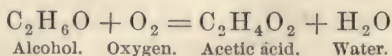
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## CHAPTER II.

### THEORY OF THE FORMATION OF VINEGAR.

INDEPENDENT of the formation of acetic acid by destructive distillation, the chemical processes by which acetic acid in larger quantities is formed are at present quite well understood, and may be briefly explained as follows :

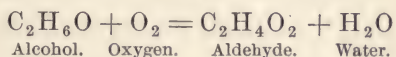
As previously mentioned, Döbereiner, in 1822, established the theory of the formation of acetic acid from alcohol, and the processes taking place thereby may be expressed by the following formula :





According to the above formula, acetic acid and water are formed by the action of oxygen upon alcohol, and hence the formation of acetic acid takes place by a partial combustion or oxidation of the latter. Alcohol and acetic acid are, however, only two members of the process, and that besides them other bodies are formed from the alcohol, can in a vinegar manufactory be readily detected by the sense of smell.

By treating alcohol with pyrolusite and sulphuric acid—hence by the action of oxygen at the moment of its liberation from a combination, *i. e.*, in its nascent state—Döbereiner obtained a body which he called “light oxygenated ether” (*leichter Sauerstoffäther*). Liebig later on studied this combination more accurately, and found that, as regards its composition, it differed from that of alcohol only by containing two atoms less of hydrogen. He applied to it the term “aldehyde.” Aldehyde is composed of  $C_2H_4O$ , and its formation is represented by the formula—



In the examination of the properties of aldehyde it was shown that it is readily converted into acetic acid by the absorption of oxygen and, based upon these facts, Liebig established a theory of the formation of vinegar which was for many years considered correct.

Essentially Liebig's theory is as follows: By the exposure, under suitable conditions, of alcohol to the action of the atmospheric oxygen, one-third of the entire quantity of hydrogen contained in it is withdrawn, and aldehyde is formed. The latter, however, immediately combines further with oxygen, and is converted into acetic acid; the formation of vinegar from alcohol being, therefore, a partial process of combustion.

From the present standpoint of our knowledge regarding the formation of acetic acid from alcohol, the correctness of this theory is about parallel with that according to which alcohol and carbonic acid are formed by the alcoholic fermentation of

sugar. The latter process can also be illustrated by an equation in as simple a manner as the conversion of alcohol into acetic acid by aldehyde. At the present time, the processes taking place in the formation of acetic acid from alcohol must, however, be considered as far more complicated than supposed by Liebig. According to the latter, a simple oxidation, *i. e.*, a simple chemical process, takes place. But, according to the now universally accepted view, the formation of vinegar is due to a chemico-physiological process with the co-operation of a living organism. Alcohol and oxygen alone do not suffice for this purpose, the presence of nitrogenous bodies and salts, besides that of an organism, being absolutely necessary.

The French chemist, Pasteur, was the first to establish the formation of vinegar as a peculiar process of fermentation, and he maintains that a certain organism, the "vinegar ferment" or "vinegar yeast," consumes the alcohol, nitrogenous substances and salts, and separates acetic acid, aldehyde, etc., as products of the change of matter taking place in the living organism. On the other hand, the German chemist Nageli is of the opinion that the role of the organism is to bring the particles of the substance to be fermented—in this case alcohol—lying next to it, into such vibrations as to decompose them into more simple combinations—in this case, acetic acid, aldehyde, etc.

The scientific dispute over these two different views is not yet settled, though the majority of chemists are inclined to accept Pasteur's theory. For the practical man it is of no consequence which of these views will be finally accepted as the correct one; the fact that the process of the formation of vinegar is connected with the living process of an organism being alone of importance to him.

As is well known, organisms producing fermentation are named after certain products which they form in larger quantities, the organisms forming alcohol from sugar being, for instance, briefly termed "alcoholic ferment." In this sense we may also speak of a vinegar or acetic ferment, since a definite

organism causing the formation of larger quantities of acetic acid from alcohol is known, and the cultivation of this ferment is one of the principal tasks of the manufacturer of vinegar.

Numerous observations have established the fact that the properties of forming large quantities of acetic acid are inherent only in this ferment. Small quantities of acetic acid are, however, also, constantly formed by other ferments, so that in examining products due to the process of decomposition induced by organisms, acetic acid will generally be found among them. In the alcoholic fermentation, at least in that of wine and bread dough, acetic acid is always found. It originates in the germination of many seeds, and generally appears in the putrefaction of substances rich in nitrogen, such as albumen, glue, etc. It appears also in the so-called lactic fermentation, the lactic acid formed by the specific ferment of this species of fermentation being by further processes of fermentation decomposed into butyric and acetic acids.

Acetic acid is found in many animal juices, for instance, in meat juices, milk, sweat and urine. It also occurs in the fresh fruit of the tamarind. The processes which take place in its formation in these cases are not known, though it is very likely directly formed from certain varieties of sugar.

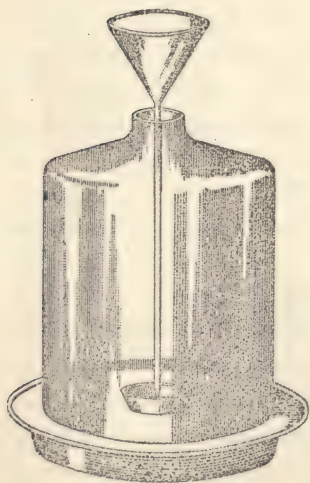
There is quite a large series of chemical processes in which certain quantities of acetic acid are formed. Sugar, starch, woody fibre and, in general, all compounds known as carbohydrates, when fused with caustic alkalies, yield certain quantities of acetic acid, and also by themselves when subjected to destructive distillation. Among the processes by which acetic acid is produced in a purely chemical manner, *i. e.*, without the co-operation of organisms, the most interesting is that by which its formation is effected by the action of very finely divided platinum, the so-called platinum black, upon alcohol. Platinum black is readily prepared by boiling a solution of platinic chloride, to which an excess of sodium carbonate and a quantity of sugar have been added, until the precipitate formed after a little time becomes perfectly black, and the



supernatant liquid colorless. The black powder is collected on a filter, washed and dried by gentle heat. On account of the minute state of its division, this substance condenses within it several hundred times its volume of oxygen, and consequently when the vapor of alcohol comes in contact with it, a supply of oxygen in a concentrated state is presented to it, and the platinum, without losing any of its inherent properties, effects chemical combination, the alcohol undergoing slow combustion and being converted into acetic acid. In order that the reaction may continue it is, of course, necessary to present fresh oxygen to the platinum to replace that which has been withdrawn. The two actions then go on side by side.

This can be illustrated by an apparatus similar to Fig. 1.

FIG. 1.



It consists of a glass bell through the mouth of which a long funnel passes. The lower end of this funnel terminates in a fine point so that the alcohol may percolate very slowly. The vessel is placed upon supports within a dish in which is a saucer or small shallow basin containing the platinum black. The interspace between the bottom of the dish and the glass bell serves for the circulation of air in the latter. A short time after the alcohol has been poured into the funnel an odor of acetic acid, arising from the acetic acid vapors which are generated, is perceived at the mouth of the bell.

These vapors condense on the walls of the bell and trickle to the bottom, where they collect in the vessel in the dish. It is of advantage, for the success of the experiment, to have the alcohol heated to about  $90^{\circ}$  F. before pouring it in. By washing and igniting the platinum used for the oxidation of the alcohol, it can be again employed for the same purpose.

Independent of the purely chemical methods which, with the exception of that by which acetic acid is produced by the destructive distillation of wood, are of no practical importance, the formation of vinegar, no matter what method may be adopted, can only be effected in the presence of certain organisms. It has long been known that organisms to which the term *mother of vinegar* has been applied, develop upon liquids containing, besides alcohol, certain other substances, for instance, upon weak wine or beer, and this mother of vinegar has also been used for making vinegar on a large scale. To Pasteur, however, belongs the incontestable merit of having more accurately examined the relations of these organisms to the formation of vinegar. These examinations gave rise to his experiments on the diseased alteration of wine, which were later on superseded by his researches on the formation of wine vinegar.

Pasteur found that upon the surface of every fluid capable, by reason of its composition, of being converted into vinegar, organisms develop immediately after the commencement of the formation of vinegar. He recognized these organisms as fungoid plants of a low order and called them *Mycoderma aceti*. More recent researches on the botanical nature of these plants show them to belong to the group of lowest fungoid organisms, to which the term *bacteria* or *schizomycetes* has been applied.

The *Bacterium aceti*, the name applied to this organism, consists of a single, generally globular or filiform cells, its special characteristic being its mode of propagation, which is effected by the division of the cell into two, and then a separation or splitting of both.

The exceedingly minute size of the schizomycetes and their great resemblance to each other make their accurate determination very difficult, and hence it is customary to name the better known species in accordance with the chemical products they form or in accordance with the phenomena they produce. Among the first kind may be classed those which effect the formation of acetic, lactic, butyric acids, while other very little

known bacteria must be considered as the cause of the so-called nitric acid fermentation, and again others appear in putrid fermentation. A special group of bacteria reaches development in animal organisms and gives rise to terrible diseases, some causing rinderpest, others tuberculosis and various other maladies. Cholera and other epidemics have been found to be due to certain bacteria.

The bacteria causing disease are of course very interesting to the physician, but to the manufacturer of vinegar a thorough knowledge of the conditions of life governing the vinegar bacteria is of the utmost importance in order to conduct the manufacture in such a manner that disturbances shall rarely occur, and should they happen, that he may be able to remove them. It may therefore be said that the entire art of the manufacture of vinegar consists in an accurate knowledge of the conditions of life of the vinegar bacteria and in the induction of these conditions of life. As long as the latter are maintained the process of the formation of vinegar will go on without disturbance, and the origination of new generations of vinegar ferment be connected with the conversion of certain quantities of alcohol into vinegar.

Pasteur regarded the bacterial growth mentioned above as consisting of a single species. Hansen, however, showed in 1878, that in the spontaneous souring of beer at least two different species of bacteria can come into action, one of which he named *Mycoderma aceti* and the other *Mycoderma Pasteurianum*. At the suggestion of W. Zopf, he afterwards changed these names to *Bacterium aceti* and *Bacterium Pasteurianum* respectively. The number of species has been further increased by recent investigations, and among these acetic acid bacteria there are several, the activity of which is distinctly different, and the employment of a pure culture of systematically selected species would be desirable in the manufacture of vinegar. Searching investigations into the chemical activity of the different species of acetic acid bacteria would be not only opportune in the interests of science, but also highly important in the practice of the vinegar industry.



## CHAPTER III.

## THE VINEGAR FERMENT AND ITS CONDITIONS OF LIFE.

A. *The Vinegar Ferment*—While but little is known about the origin of the vinegar ferment, experiments have shown these organisms to be everywhere distributed throughout the air and to multiply at an enormous rate when fluids of a composition suitable for their nutriment are presented to them. A fluid especially adapted for this purpose is, for instance, thoroughly fermented ripe wine, its exposure in a shallow vessel at the ordinary temperature of a room being sufficient to induce the propagation of the vinegar bacteria reaching it from the air.

The experiment is, however, certain of success only when made with ripe wine, by which is meant wine which shows but little turbidity when vigorously shaken in contact with air and exposed in a half-filled bottle to the air. Young wine contains a large quantity of albuminous substances in solution, and is especially adapted for the nutriment of an organism, the *saccharomyces mesembryanthemum* belonging to the saccharomycetes. It develops upon the surface of such wine as a thick white skin, which later on becomes wrinkled and prevents the growth of the vinegar ferment. A fluid well adapted for the nutriment of the vinegar ferment and which may be substituted for wine for its culture is obtained by adding 5 to 6 per cent. of alcohol and about  $\frac{1}{2}$  per cent. of malt extract to water.

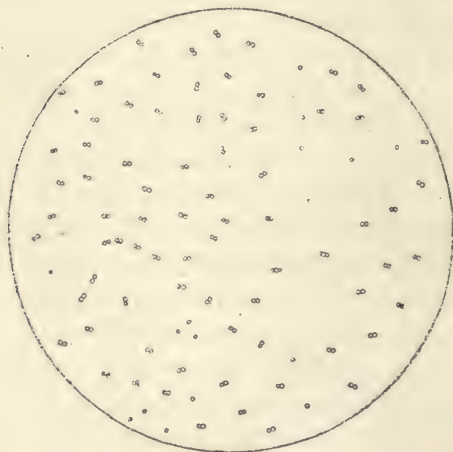
By exposing this fluid, or ripe wine at the ordinary temperature of a room, best in a dish covered by a glass plate resting upon small wooden blocks to prevent the access of dust, the formation of a thin veil-like coating upon the surface will in a few days be observed. The wine soon exhibits the characteristic odor and taste of acetic acid, and in a few days assumes a somewhat darker color, and deposits a slight brownish sediment consisting of decayed vinegar ferment. In 14 to 21 days

the fluid is entirely converted into vinegar, *i. e.*, it contains no more alcohol, but in place of it the corresponding quantity of acetic acid.

By exposing the vinegar thus obtained for a longer time to the air, a thick white skin of mold may happen to form on the surface, and on testing the fluid, it will be found that the content of acetic acid steadily decreases, the mold which is able to convert the alcohol into water and carbonic acid possessing also the power of forming the same products from acetic acid.

The process above described of the destruction of the wine

FIG. 2



and its conversion into vinegar by a veil-like coating of vinegar ferment occurs most frequently, though a thick spume, the so-called *mother of vinegar*, may also happen upon the surface. This phenomenon will be referred to later on.

On examining under the microscope a drop taken from the surface of the wine when the veil of vinegar ferment commences to form, a picture like that shown in Fig. 2 presents itself. In a somewhat more advanced stage, formations resembling chains and strings of beads appear more frequently, and when finally the development of the ferment is in full

progress, it appears as an aggregation of numerous single cells mixed with double cells and many other cells strung together like beads. The field of vision of the microscope is then completely filled with a large number of colorless globules, which are present either singly or in combinations of two, formations resembling chains or strings of beads being of rare occurrence. In many of the separately-occurring formations oval forms, generally slightly contracted in the centre, are observed, this contraction indicating the place where the splitting of one cell into two new cells takes place. By vigorously shaking the fluid before viewing it under the microscope, very few of the above mentioned bead-like formations will be found, but more frequently the contracted ones. By observing for hours a drop of the fluid containing the ferment in an advanced state of development, the globules strung together will be noticed to fall apart when at rest. Hence it may be supposed that in the propagation of cells by splitting, the newly formed cells adhere together up to a certain stage, and later on separate in the fluid when in a quiescent state. Like every other organism the vinegar ferment only lives for a certain time, and after dying sinks below the fluid and forms upon the bottom of the vessel the above-mentioned sediment. The latter appears under the microscope in the same form as the living ferment, but differs from it in being less transparent, and of a brownish color. The propagation of the vinegar ferment takes place very rapidly, and it will be found in a few hours after the commencement of its development in all stages of life upon the surface of the fluid, it being possible to distinguish cells of from 1.5 to 3.5 micromillimeters.\*

The vinegar ferment requiring free oxygen for its propagation, grows exuberantly only upon the surface of the nutrient fluids. By filling a bottle about four fifths full with wine, and after allowing the vinegar ferment to develop, closing the mouth of the bottle with the hand, and submerging the neck of the

\*.One micromillimeter =  $\frac{1}{1000}$  millimeter.



bottle in water, the fluid will be seen to rise for some time in the bottle, and then remain stationary. A determination of the content of acetic acid immediately before the commencement of this experiment, and a few days after, shows but a slight increase in acetic acid, because the ferment has consumed the free oxygen present in the bottle, the essential condition for its further development is wanting, and it must cease its activity, without, however, perishing. It may here be remarked that the vinegar ferment, like the majority of bacteria, possesses an extraordinary vitality. Under unfavorable conditions it passes into a kind of quiescent state, during which no perceptible increase of cells takes place, and it may remain in this state for a long time without suffering destruction, but as soon as the conditions for its nutriment are again presented, propagation in a normal manner recommences.

The great rapidity of propagation of the vinegar bacteria is shown by an experiment of some importance to the practice. Pour into a shallow vat, about three feet in diameter, a fluid suitable for the nutriment of the bacteria, and divide upon the surface by means of a thin glass rod small drops of wine, upon which the frequently mentioned veil has been formed. In a few hours the entire surface of the fluid in the vat will be covered with vinegar bacteria, spreading concentrically from the points where the drops of wine have been distributed. From this it will be seen that the culture of the ferment for the purpose of manufacturing vinegar offers no difficulties, provided all conditions for its propagation be observed.

*B. Conditions for the Nutriment of the Vinegar Ferment.* The conditions most favorable for the development of the vinegar ferment, and for converting in the shortest time the largest quantity of alcohol into acetic acid, have been determined by many observations and long experience in the practice. These conditions will first be briefly enumerated, and then the separate points more fully discussed.

For the vinegar bacteria to settle upon a fluid, and for their vigorous propagation, the following factors are required :

1. A fluid which, besides alcohol and water, contains nitrogenous bodies and alkaline salts. The quantities of these bodies must, however, not exceed a certain limit.
2. The fluid must be in immediate contact with oxygen (atmospheric air).
3. The temperature of the fluid and the air surrounding it must be within certain limits.

As regards the composition of the nutrient fluid itself, it must contain all the substances required for the nutriment of a plant of a low order, such substances being carbohydrates, albuminates and salts. Alcohol must be named as a specific nutriment of the vinegar ferment, provided the supposition that the latter consumes the alcohol and separates in its place acetic acid, is correct. The quantity of alcohol in the fluid intended for making vinegar must, however, not exceed a certain limit, a content of 15 per cent. appearing to be the maximum at which acetic fermentation can be induced. But even a content of 12 to 13 per cent. of alcohol is not very favorable for the vegetation of the vinegar ferment, and every manufacturer knows the difficulty of preparing vinegar from such a fluid. A small quantity of acetic acid in the nutrient medium exerts also an injurious influence upon the vinegar ferment. Upon a fluid containing 12 to 13 per cent. of acetic acid and 1 to 2 per cent. of alcohol, the ferment vegetates only in a sluggish manner, and considerable time is required to convert this small quantity of alcohol into acetic acid.

That the vinegar ferment cannot live in dilute alcohol alone may be shown by a simple experiment. By impregnating a fluid consisting only of water and alcohol, a very small quantity of acetic acid is formed to be sure, but the ferment perishes in a short time—it starves to death. A fluid suitable for its nourishment must, therefore, contain the above-mentioned nutrient substances, sugar, dextrine, or similar combinations occurring in wine, malt extract, and beer, being generally employed as carbohydrates. These fluids further contain nitrogenous combinations which may serve as nutrient for the fer-

ment, also considerable quantities of phosphates. Hence, by an addition of wine, malt extract, beer, or any fruit wine (apple or pear cider) to a mixture of alcohol and water, a fluid can be prepared that contains all the substances essential to the nutriment of the ferment.

The necessary quantity of these nutrient substances, as compared with that of alcohol, is very small, since the quantity by weight of vinegar ferment required for the conversion of a very large amount of alcohol into vinegar is only a few fractions of one per cent. of the weight of alcohol used. Hence the manufacturer may be very sparing with the addition of nutrient substances to the fluid to be converted into vinegar, without having to fear that the ferment will be stunted.

The vinegar ferment is very sensitive to sudden changes in the composition of the fluids upon which it lives, and suffers injury by such changes which are recognized by diminished propagation and a decrease in the conversion of alcohol into acetic acid.

By transferring, for instance, vinegar ferment which had normally vegetated upon a fluid containing only 4 to 5 per cent. of alcohol, to one with a content of 10 to 11 per cent., its propagation, as well as its fermenting energy, decreases rapidly and remains sluggish, until a few new generations of cells have been formed which are better accustomed to the changed conditions. By bringing, on the other hand, a ferment from a fluid rich in alcohol upon one containing a smaller percentage, the disturbances in the conditions of the ferment can also be observed, but they exert a less injurious influence upon the process of the formation of vinegar than in the former instance.

The process of nutriment of the vinegar ferment, however, must not be understood to consist simply in the consumption of sugar, albuminates and salts. It differs according to the composition of the nutrient medium, and is so complicated as to require very thorough study for its explanation. If, for instance, wine is converted into vinegar, and the composition of the latter compared with that of the original wine, it will be



found that not only the alcohol has been converted into acetic acid and the fluid has suffered a small diminution of extractive substances and salts, which might be set down to the account of the nutriment of the ferment, but that the quantity of tartaric, malic and succinic acids has also decreased, as well as that of glycerine, and of the latter even nothing may be present. Hence it must be supposed that the vinegar ferment derives nutriment also from these substances, or that the fermenting activity acts upon them as well as upon the alcohol. There is finally the fact of great importance for the practice, but which has not yet been sufficiently explained, that the vinegar ferment develops more rapidly upon a fluid which, besides the requisite nutrient substances, contains a certain quantity of acetic acid, than upon a fluid entirely destitute of it. Regarding the supply of air, it may be said that, while for mere existence the vinegar ferment requires comparatively little air, large quantities of it are necessary for its vigorous propagation and fermenting activity. In the practice it is aimed to accomplish this by exposing the fluid in which the ferment lives in thin layers to the action of the air, and, in fact, upon this the entire process of the quick method of manufacture is based.

Besides the above-mentioned factors, the temperature to which the ferment is exposed is of great importance as regards its development. The limits at which the propagation of the ferment and its vinegar-forming activity are greatest, lie between  $68^{\circ}$  and  $95^{\circ}$  F. Above this limit the formation of vinegar decreases rapidly, and ceases entirely at  $104^{\circ}$  F. However, when the temperature is again reduced to  $86^{\circ}$  F., the ferment reassumes its activity. At a temperature exceeding  $104^{\circ}$  F. the ferment suffers perceptible injury; heated to  $103^{\circ}$  F. it becomes sensibly weaker, and at first propagates very slowly, regaining its original vigorous development only after several generations. When the temperature of the fluid is raised to  $122^{\circ}$  F. the ferment perishes.

The ferment appears to be less affected by low temperatures.

If the temperature of a fluid which shows an exuberant vegetation of ferment is reduced to  $50^{\circ}$  F., the formation of vinegar continues, though at a much reduced rate. Special experiments have shown that when wine with a vegetation of ferment is converted into ice by being exposed to a temperature of  $14^{\circ}$  F., and then melted and heated to  $59^{\circ}$  F., the ferment recommences to grow and to form acetic acid. It must, however, be remarked that while vinegar ferment in a state of development keeps up a slow vegetation when the fluid is reduced to a low temperature, it is extremely difficult to cultivate it upon a cold fluid. This is very likely the reason why acetic degeneration is not known in cold wine cellars, while in cellars with a temperature of over  $59^{\circ}$  F., this dreaded process can only be guarded against by the greatest care.

Since the propagation of the ferment and its fermenting activity increase with a higher temperature, it would appear most suitable to keep the temperature of the fluid to be converted into vinegar as near the uppermost limit of  $95^{\circ}$  F. as possible. Experience, however, has shown that at this temperature disturbances are of frequent occurrence in the generators, and for this reason one of  $86^{\circ}$  to  $89^{\circ}$  F. is generally preferred. The process of the formation of vinegar itself explains why disturbances may easily occur at a high temperature. It is a chemical (oxidizing) process in which a certain quantity of heat, depending on the quantity of alcohol to be oxidized within a certain time, is always liberated. If now by the use of a temperature close to  $95^{\circ}$  F., the activity of the ferment is strained to the utmost, a large quantity of alcohol is in a short time converted into acetic acid, and consequently so much heat is liberated that the temperature in the generator rises above the permissible maximum and the ferment immediately ceases its activity. Thus it may happen that in a generator which has satisfactorily worked for some time, the formation of vinegar ceases all at once, and on examining the thermometer placed on the apparatus the cause will be generally found to be due to too high a temperature.

*Mother of Vinegar*—In connection with the description of the conditions of life of the vinegar bacteria, a peculiar formation, playing in many cases a rôle in the practice of making vinegar, has to be mentioned. This is the so-called mother of vinegar, the term having very likely been applied to it on account of its causing acetification when brought into a fluid suitable for the formation of acetic acid. The first botanical investigation of this substance was made in 1822 by Persoon, who described the organized skin developing on various fluids, and gave it the general name of *Mycoderma*, i. e., mucinous skin or fungoid skin.

Kützing, in 1837, showed that the "mother of vinegar" is constructed of a number of minute dot-like organisms—which are now called bacteria—arranged together in the form of chains. These he classified as algae, and named them *Ulvina aceti*, and asserted quite positively that alcohol is converted into acetic acid by the vital activity of these organisms. Kützing's results, however, attracted but little attention because two years after their publication, Liebig appeared on the scene with his theory of acetic fermentation, which has already been referred to, in which no mention was made of the potency of living organisms, but the "mother of vinegar" was asserted to be a formation devoid of life, a structureless precipitate of albuminous matter. One of the reasons put forward in support of this view was a chemical analysis of the "mother of vinegar" by the Dutch chemist, G. Mulder, who because he failed to discover the presence of any ash constituents, thought that it must be regarded as a compound of protein and cellulose. Mulder's statement was refuted in 1852 by R. Thomson, who showed that a sample of "mother of vinegar" contained 94.33 per cent. water, 5.134 per cent. organic matter and 0.336 per cent. ash.

The formation of mother of vinegar can always be successfully attained by exposing young wine to the air until the commencement of the formation of mold is indicated by the appearance of white dots and then transferring the wine to a



room having a temperature of  $86^{\circ}$  F. At this temperature the development of the vinegar ferment proceeds so vigorously that it suppresses the mold ferment, and the peculiar mass constituting the mother of vinegar soon forms upon the surface.

Mother of vinegar occurs so generally in young wine which is largely used for the preparation of wine vinegar, that its formation was considered as inseparably connected with that of acetic acid from alcohol, while actually it is only due to the peculiar constitution of the fluid to be converted into vinegar. In many places this opinion is still entertained, and especially where, as is generally the case, the manufacture of vinegar from wine is yet carried on in the primitive way of centuries ago. In speaking of the preparation of vinegar from wine, it will be shown that the conversion can be effected by means of the ordinary vinegar ferment without the appearance of mother of vinegar.

#### *Summary.*

Briefly stated, the points of the theoretical conditions of the formation of vinegar of importance to the manufacturer are:—

1. Acetic acid is formed during many chemical conversions.  
However, for the manufacture of acetic acid, and consequently of vinegar on a large scale, only two methods are available, viz., the preparation of vinegar from alcohol by fermentation, or the production of acetic acid by the destructive distillation of wood.
2. All alcoholic fluids formed by vinous fermentation of sacchariferous plant juices or fermented malt extracts are suitable for the preparation of vinegar by fermentation. Specially prepared mixtures of water, alcohol and vinegar may also be used for the purpose, provided they contain small quantities of certain organic substances and salts, and not over 14 per cent of alcohol.
3. Acetic fermentation is induced by a microscopic organism belonging to the bacteria, and the conversion of the alcohol into acetic acid is in a certain ratio to the propagation of this organism.

4. Besides the substances mentioned in 2, the vinegar ferment requires for its vigorous development free oxygen and a temperature lying between  $68^{\circ}$  and  $95^{\circ}$  F.
  5. In the acetic fermentation the greater portion of the alcohol is converted into acetic acid and water; besides these, small quantities of other products are formed which are in a measure not yet thoroughly known. In the conversion of wine, beer, etc., other combinations contained in the fluids, besides alcohol, are also essentially changed.
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## CHAPTER IV.

### PRODUCTS OF ACETIC FERMENTATION.

THE formation of vinegar by fermentation being a chemico-physiological process, many and complicated chemical processes must take place in the fluid to be converted into vinegar in order to produce all the combinations required for the propagation of the ferment. Attention cannot be too frequently called to the fact that from the standpoint of the manufacturer, the regular propagation of the ferment is the main point of the entire manufacture, the quick conversion of the alcohol contained in the fluid being a necessary consequence of it.

The body of the ferment, however, contains cellulose, albuminous substances, very likely fat and other combinations not yet known, all of which must be formed from the nutrient substances (sugar, dextrine, albuminous substances, etc.), present. It being very probable that a portion of the alcohol contained in the fluid is consumed for this purpose, a small but nevertheless perceptible loss of alcohol will occur in the production. It would be erroneous to suppose that the conversion of alcohol into acetic acid and water is effected accord-

ing to the formula given on p. 6, since a certain portion of it is always converted into other combinations, the nature and formation of which can only be, to a certain extent, explained.

In the vinous fermentation, which of all fermenting processes has been most thoroughly studied, it is found that from the sugar, besides alcohol and carbonic acid, large quantities of glycerine and succinic acid and probably other bodies are formed, which must undoubtedly be classed among the products of vinous fermentation. Similar processes, no doubt, take place in acetic fermentation, and besides acetic acid and water other little-known products of fermentation are regularly formed.

According to the nature of the sacchariferous fluids subjected to vinous fermentation, small quantities of certain bodies called fusel oils are formed which are decidedly products of fermentation. They impart to the fermented fluid, as well as to the alcohol distilled from it, such characteristic properties that from the odor of the alcohol a correct judgment can be formed as to the material employed in its preparation.

In the conversion of such a fluid, or of alcohol prepared from it, into vinegar, the fusel oils are also changed—very likely oxydized—and with some experience the material (wine, beer, malt, etc.), from which the vinegar has been made can be determined by the sense of smell. The quantities of aromatic substances which reach the vinegar in this manner are, of course, very small, but they must nevertheless be classed among the most important products of acetic fermentation, they being characteristic as regards the derivation of the vinegar. Of the products of acetic fermentation, besides acetic acid, aldehyde and acetal are best known, these combinations appearing always, even in small quantities, in making vinegar according to the methods customary at the present time.

*Acetic Aldehyde or Acetaldehyde*, commonly called simply aldehyde (from *alcohol dehydrogenatum*), is obtained by oxidizing spirits of wine by means of manganese dioxide (pyrolu-



site) and sulphuric acid, or platinum black, in the presence of air, or if alcohol or ether be burning without a sufficient supply of air. It is also formed by heating a mixture of calcium acetate and calcium formate. It is contained in considerable quantities in the first runnings obtained in the manufacture of spirit of wine.

To prepare pure aldehyde, 3 parts of potassium dichromate in small pieces are placed in a flask surrounded by a freezing mixture and a well-cooled mixture of 2 parts of spirit of wine, 4 of sulphuric acid, and 4 of water added. After connecting the flask with a condenser the freezing mixture is removed; a violent reaction soon sets in, and the liquid begins to boil. The vapors have first to pass through an ascending tube surrounded by warm water at about  $122^{\circ}$  F. Alcohol and various other products are condensed and flow back, while the vapor of the aldehyde, after having passed through a descending condenser, is absorbed in anhydrous ether.

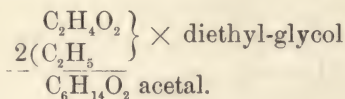
Pure aldehyde thus obtained is a colorless liquid of the composition  $C_2H_4O$ . Its specific gravity is 0.800, and it boils at about  $71.5^{\circ}$  F. It has a pungent and suffocating odor, and is readily soluble in water, alcohol and acetic acid. Like all the aldehydes it is very easily oxidized and acts, therefore, as a powerful reducing agent. Thus, on heating it with a little ammonia and nitrate of silver, metallic silver separates out, coating the sides of the vessel with a bright mirror. It combines with ammonia, and forms a crystalline compound which has the peculiar odor of mice.

Though it is likely that in the manufacture of vinegar by the quick process, besides aldehyde, acetic and formic ethers are formed, they are of comparatively little importance for our purposes. Of more importance, however, is acetal, the formation of this combination affording an interesting insight into acetic acid.

*Acetal* is best prepared by distributing pieces of pumice, previously moistened with 25 per cent. alcohol, over a large glass plate, placing watch crystals containing platinum black

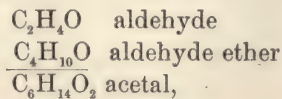
upon the pieces of pumice, and covering the whole with a large bell-glass. The alcohol absorbed by the pumice being converted into acetic acid, 60 per cent. alcohol is poured upon the plate and the air in the bell-glass from time to time renewed. In a few weeks quite a thick fluid of an agreeable odor has collected upon the glass plate. This is collected and distilled, the portion passing over at  $219^{\circ}$  F. being collected by itself.

Pure acetal is composed of  $C_6H_{14}O_2$ . It is a colorless liquid, has a specific gravity of 0.821, and boils at  $219.2^{\circ}$  F. It has a refreshing odor, calling to mind that of fruit ethers. By oxidizing agents it is rapidly converted into acetic acid. Nitrate of silver in the presence of ammonia, however, is not reduced by it, and it remains unchanged on boiling with potash lye. From its composition acetal may be considered from several points of view. It may be regarded as an ethyl alcohol (glycol)  $C_2H_6O_2$ , in which two atoms of hydrogen have been replaced by two molecules of the radical ethyl  $C_2H_5$ , hence thus

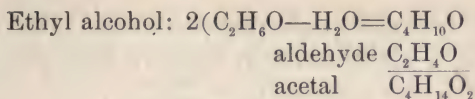


This view of the composition of acetal is supported by the fact that methyl or amyl can be substituted for either one or both molecules of ethyl in the combination.

According to other opinions, acetal may be considered as a combination of aldehyde and aldehyde ether :—



or as a combination of aldehyde with ethyl alcohol, one molecule of water in the latter having been replaced by the aldehyde :—



By keeping in view the fact that the process of the formation of vinegar is an oxidation of the alcohol which does not proceed with equal energy in all parts of the apparatus, it will be understood that during this process aldehyde, acetal, and acetic ether may be formed which, if the operation be correctly conducted, will be finally converted into acetic acid, though small quantities of them will be found in the vinegar when just finished and exert an influence upon its constitution.

*Pure acetic acid*,  $\text{C}_2\text{H}_4\text{O}_2$ , cannot be directly obtained from vinegar, but only from acetates by methods which will be described later on. The strongest acetic acid which can be prepared is known as *glacial acetic acid*, from its crystallizing in icy leaflets at about  $40^\circ \text{F}$ . Above about  $60^\circ \text{F}$ . the crystals fuse to a thin, colorless liquid of an exceedingly pungent and well-known odor. Pure acetic acid is a powerful restorative when applied to the nostrils in impending fainting. It is the strongest of organic acids and nearly as corrosive as sulphuric acid. Applied to the human skin it acts as an irritant, causing redness and swelling, followed by paleness of the part, and, if its application be prolonged, it is followed by vesication and desquamation of the cuticle. It first whitens mucous membranes, then turns them brown, causing meanwhile a severe burning pain. Highly concentrated acetic acid is a solvent of many volatile oils and resins, and in practice its high concentration is tested by its ability to dissolve lemon oil, since in the presence of only 2 per cent. of water in the acid, lemon oil is no longer dissolved by it.

The specific gravity of pure acetic acid is at  $59^\circ \text{F}$ .:—

According to Oudemans . . . . .	1.0553
“ Roscoe . . . . .	1.0564
“ Kopp . . . . .	1.0590
“ Mendelejeff . . . . .	1.0607
“ Mohr . . . . .	1.0600



According to Mohr's determinations, the specific gravity of pure acetic acid varies much at different temperatures, it being

1.0630	at	54.5° F.
1.0600	"	59.0 "
1.0555	"	68.0 "
1.0498	"	77.0 "
1.0480	"	79.0 "

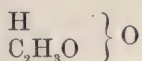
Mixtures of acetic acid and water show a peculiar behavior in regard to their specific gravity, the latter rising steadily until the content of water amounts to from 20 to 23 per cent. The density of the liquid then diminishes so that a mixture containing 46 per cent. of water shows the same specific gravity as the anhydrous acid. From this point on, the specific gravities of the mixtures decrease with the increase in the content of water.

This peculiar behavior of the mixtures renders the accurate determination of the content of acid in a concentrated mixture, by means of the aerometer, impossible. There are a number of determinations of specific gravities of acetic acid with varying contents of water (by Mohr, von der Toorn, Oudemans, etc.), but they differ considerably from each other, like the tables at the end of this volume, so that, while the specific gravity test answers very well for the determination of the amount of anhydrous acid in dilute solutions, it is very fallacious when the acid increases in strength, and an accurate determination can only be effected by chemical methods.

Highly concentrated acetic acid has found considerable application in photography and surgery, and frequently occurs in commerce in the form of so-called *vinegar essence*. The acetic acid occurring under this name is generally prepared from wood vinegar, and is only fit for the preparation of table vinegar when a chemical examination shows no trace of tar products, which, besides acetic acid, are formed in abundance in the destructive distillation of wood.

In regard to the composition of acetic acid, it may be mentioned that one atom of hydrogen can be readily replaced by

univalent metals or univalent compound radicals which may be expressed by



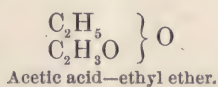
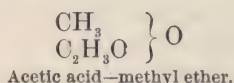
whereby the acetic acid is considered as water  $\begin{array}{c} \text{H} \\ \text{H} \end{array} \} \text{O}$  in which one atom of hydrogen is replaced by the compound radical  $\text{C}_2\text{H}_3\text{O} = \text{acetyl}$ .

If the one atom of hydrogen standing by itself be replaced by a univalent metal, a neutral acetate is formed, for instance :

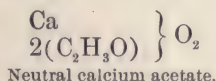


or sodium acetate.

If this atom of hydrogen is replaced by a univalent compound radical, for instance, by methyl  $\text{CH}_3$ , or ethyl  $\text{C}_2\text{H}_5$ , the so-called compound ethers are formed.



If a bivalent metal or compound radical yields a neutral combination with acetic acid, the substituted hydrogen in two molecules of acetic acid must evidently be replaced by this bivalent metal, for instance :—



*Theoretical Yields of Acetic Acid*—In industries based upon chemical processes a distinction is made between the *theoretical* and *practical* yields.

By *theoretical* yield is understood the quantity of the body to be manufactured which would result if no losses of substance were connected with the chemical process; the *practical* yield, on the other hand, is that in which such losses are taken into account, the average being ascertained by long-continued

comparison of daily yields. The closer the practical yield approaches the theoretical one, the more suitable the method pursued in the production evidently is, and thus the manufacturer, who has a clear idea of the theoretical yield, can readily judge of the value of his method by comparing it with the practical yield attained.

Now suppose no loss of substance (by evaporation or formation of other combinations) occurs in the conversion of alcohol into acetic acid, it can be readily calculated from the composition of the two bodies how many parts by weight of acetic acid can be formed from a determined number of parts by weight of alcohol.

Alcohol has the composition  $C_2H_6O$ , or an atomic weight of 46, because :—

$C_2 =$	.	.	.	24
$H_6 =$	.	.	.	6
$O =$	.	.	.	16
				—
Make .	.	.	.	46

The composition of acetic acid is  $C_2H_4O_2$  and its molecular weight 60, because :

$C_2 =$	.	.	.	24
$H_4 =$	.	.	.	4
$O_2 =$	.	.	.	32
				—
Make .	.	.	.	60

Hence from 46 parts by weight of alcohol 60 parts by weight of acetic acid may be formed, or by taking 100 parts of alcohol it follows that 100 parts by weight of alcohol must yield 130.43478 parts by weight of acetic acid. This increase in weight has to be attributed to the absorption of one atom of oxygen, atomic weight 16, against the loss of two atoms of hydrogen, atomic weight 2. Since these two atoms of hydrogen are themselves oxidized to water by the absorp-



tion of oxygen, the total yield from 100 parts by weight of alcohol would be :

Acetic acid . . . . .	130.43478	parts by weight.
Water . . . . .	39.13043	“ “ “
Total . . . . .	169.56521	parts by weight.

The quantity of oxygen required to form acetic acid and water from 46 parts by weight of alcohol, amounts to 32 parts by weight, hence for 100 parts to 69.562 parts by weight. The oxygen is conducted to the alcohol in the form of air, and it can be readily calculated how much of the latter is required to convert a given quantity of alcohol, for instance, 100 grammes, into acetic acid. In round numbers the air contains in 100 parts by weight 23 parts by weight of oxygen. Since 1 liter of air of 68° F., *i. e.*, of that temperature which should at the least always prevail in the vinegar generators, weighs 1.283 grammes, the oxygen contained in it weighs 0.29509 grammes. Since, as above stated, 69.562 parts by weight are necessary for the conversion of 100 parts by weight of alcohol into acetic acid, it follows that 235.70 liters of air are required for the same purpose.

Examinations as to the content of oxygen in the air escaping from well-conducted vinegar generators have shown that on an average only one-quarter of the entire content of oxygen is consumed in the formation of vinegar, hence four times the theoretically calculated quantity of air must pass through the apparatus to completely convert the alcohol into acetic acid. Hence 100 grammes of alcohol require at least 942.92 liters of air for their conversion into acetic acid, and, without being far wrong, it may be assumed that in a vinegar factory, in round numbers, 1000 liters, or one cubic metre of air, are required for every 100 grammes of alcohol to be converted into acetic acid.

A vinegar generator, on an average, converts daily 3 litres of alcohol into acetic acid ; 3 litres of absolute alcohol (specific

gravity 0.794) weigh 2382 grammes. Now, if, as stated above, 1 cubic metre of air is required for every 100 grammes of alcohol, it follows that 23.82 cubic metres, or 23,820 liters of air must pass daily through each vinegar generator in operation. \*

Calculated to 16 working hours a day, somewhat more than 0.4 liter (more accurately 0.413 liter) must pass every second through the generator in order to supply the quantity of oxygen required for the conversion of alcohol into acetic acid.

Since the formation of vinegar has theoretically to be considered as a process of combustion, in which of 46 parts by weight of alcohol, 2 parts by weight of hydrogen, or of 100 parts by weight of alcohol 4.34782 parts by weight of hydrogen, are consumed, the quantity of heat liberated by the conversion of 100 parts by weight of alcohol into acetic acid can also be calculated. By combustion, 1 gramme of hydrogen yields 34.126 units of heat, and hence 4.34782 grammes of hydrogen, 148.373 units of heat, *i. e.*, in the conversion of 100 grammes of alcohol into acetic acid sufficient heat is liberated to heat 148.373 kilogrammes of water from 0° C. to 1° C., or 1.48 kilogrammes from 0° C. to boiling, and thus a considerable development of heat is caused by the rise of temperature in the apparatus, in which a vigorous formation of vinegar takes place.

In answer to the question, what can the practical manufacturer of vinegar learn from these theoretical explanations, it may be said there are many points of great importance for the execution of the work. The calculation of air shows that the alcohol requires a large supply ; but the generators in general use in the quick process are by no means so arranged as to be adequate to the theoretical demands. In fact it may be said that most of them allow only a limited change of air and con-

\* It is always supposed that the manufacture of vinegar is effected in generators used in the quick process.

sequently work slower than they actually should. That the generators now in use are deficient is conclusively proved by the numerous constructions which have been proposed, especially in modern times, whose chief aim is to afford a free passage to the air.

The fact that considerable heat is developed in the interior of the generator deserves consideration in connection with the heating of the manufactory. If the temperature in the latter is so high as nearly to approach the *acme*, *i. e.*, the temperature most favorable for the formation of vinegar, it may easily happen that, in consequence of the vigorous oxidation of the alcohol, the temperature in the interior of the generators be increased to such an extent as to exceed this *acme*, and the activity of the vinegar ferment would immediately diminish and even cease altogether.

If, on the other hand, the temperature of the workroom is kept too low, the generators act sluggishly and do not produce so much as when the correct conditions are observed. But while by raising the temperature of the workroom the activity of the generators is increased, too low a temperature is less injurious to the regular course of the process than too high a one.

The *acme* of the formation of vinegar is at about 86° F., and hence the aim should be to maintain this temperature as nearly as possible in the *interior* of the generator. The temperature of the *workroom* must, however, be kept sufficiently low, so that the *acme* in the interior of the generator may not be exceeded.

Another factor may here be mentioned. The closer the temperature in the interior of the generator approaches the *acme* and the quicker the supply of air, the more alcohol and acetic acid are lost by evaporation, or in other words, the smaller the yield of acetic acid. By the skillful utilization of conditions the manufacturer must aim to reduce this loss to a minimum, and this can be best effected by a suitable arrangement of the workroom. By regulating the change of air so



that it is not greater than absolutely necessary, the air will soon become so saturated with vapors of alcohol and acetic acid that no further loss will take place until the renewing of the air in the workroom appears necessary. In which manner the manufacturer is to work in order to carry on the business most advantageously depends on the conditions of trade. If large orders have to be filled, he will endeavor to increase the capacity of the generators to the utmost by maintaining the acme of temperature and a vigorous change of air in them, and in this case must submit to the increased losses inseparably connected with this high performance. If, on the other hand, he works for stock, he will not force the capacity of the generators to the utmost, but in order to work as cheaply as possible direct his attention to reduce the losses to a minimum.

*Yields of Acetic Acid Obtained in the Practice*—By keeping for some time an accurate account of the actual yields and comparing them with those theoretically obtainable, the former will be found to fall more or less short of the latter, and the difference will be the smaller, the better the method of production in use.

In a vinegar factory occur many unavoidable losses, the sources of which have been indicated in the preceding explanations; alcohol and acetic acid evaporate, and further a portion of them is entirely destroyed by too much oxidation. Now a loss by evaporation, etc., of ten per cent. of the quantity of alcohol originally used must no doubt be considered a large one; but from numerous observations it may be asserted that even with the greatest care in working, the loss in some vinegar factories is not less than from 15 to 20 per cent., and may even be as much as 30 per cent.

These enormous losses of material conclusively prove the defectiveness of the processes in general use and the urgent necessity for reformation. The experiments made for this purpose, and which have been especially directed towards a remodeling of the apparatus used, cannot be considered entirely satisfactory, though they were partially instituted by

practical manufacturers, who, however, lacked the necessary theoretical knowledge.

The principal requirement is to provide the generator with a suitable ventilator, which will allow of the passage of exactly the quantity of air required for the conversion of the alcohol into acetic acid, and is so constructed that the vapors of alcohol and acetic acid (or at least the larger portion) carried away by the current of air are condensed and thus regained.

A vinegar generator has frequently been compared to a furnace, and in continuation of this comparison it may be said, that the construction generally used is a furnace lacking every arrangement for the regulation of combustion. In such a furnace as much fuel is burned as corresponds to the quantity of oxygen entering, while in a furnace of suitable construction the combustion of fuel can be accurately regulated by increasing or decreasing at will the supply of air by means of a simple contrivance.

A vinegar generator of suitable construction should be provided with a similar arrangement. If the thermometer on the apparatus shows too low a temperature—hence too slow a process of oxidation—the course of the operation can in a short time be accelerated by the production of a stronger current of air, and the temperature correspondingly increased. If, on the other hand, oxidation proceeds too rapidly, which on account of the high temperature then prevailing in the apparatus is accompanied by considerable loss of substance, it can be quickly reduced to within the correct limits by decreasing the current of air. An apparatus unprovided with a ventilator is left more or less to itself, while one provided with such an arrangement is under the entire control of the manufacturer.

## CHAPTER V.

## METHODS OF MANUFACTURE OF VINEGAR.

FROM what has been previously said, two methods of manufacturing vinegar can only be distinguished, namely, by fermentation and by destructive distillation. It has, however, been deemed advisable to describe separately the old or slow process by fermentation and the new or quick process. The various methods employed for the manufacture of vinegar may therefore be designated as follows :

1. By fermentation according to the old or slow process.
2. By the quick process, or manufacture by fermentation with the application of improved methods in keeping with our present knowledge of chemistry.
3. Manufacture of wood vinegar, or the preparation of acetic acid by destructive distillation.
4. The preparation of pure acetic acid from acetates.

It would seem proper to commence the description of the manufacture of vinegar with the old or slow process, but for reasons of an entirely practical nature, it has been concluded not to do so, and the quick process will be first considered.

Since alcoholic fluids, directly formed by the vinous fermentation of sacchariferous plant juices, possess the property of changing, under circumstances favorable to acetic fermentation, into vinegar, it is evident that the latter can be prepared from them and, in fact, it is possible to prepare it from all sweet fruits and parts of plants, such as cherries, strawberries, figs, bananas, etc., as well as from the juices of the sugar cane, beet, chicory root, etc.

Honey, which represents a concentrated solution of fermentable sugar, as well as crystallized cane sugar, can likewise be indirectly used for the preparation of vinegar, since solutions of either can be brought into vinous fermentation, and the resulting alcohol converted into acetic acid.



By malting grain, a peculiar body called *diastase* is formed, which possesses the property of converting starch into fermentable sugar, and upon this fact is based the manufacture of beer and alcohol. In an indirect manner—the starch having to be converted first into sugar, and the latter into alcohol—it is therefore possible to prepare vinegar from every substance containing starch, and for this reason, we can speak of grain and malt vinegars. The beer prepared from the malt contains a certain quantity of alcohol, and can thus be directly converted into vinegar.

Alcohol forming ultimately the material for the manufacture of vinegar, the direct use of dilute alcohol became obvious. By the employment of a suitable process, *i. e.*, one corresponding to the laws of acetic fermentation, it was found that the conversion of dilute alcohol into acetic acid could be effected in a much shorter time than by the old method, and upon this process is based the quick process now in general use. Hence, as previously stated, two principal methods of manufacture may be distinguished, *viz.*: the old or slow process, which requires more time, and the new, or quick process.

In the old process many modifications are found, which are partially based upon old usage and partially upon the difference in the chemical composition of the raw material used. Beer, for instance, which contains only about 4 per cent. of alcohol and a large quantity of extractive substances (sugar, dextrin, salts, etc.), requires a different treatment from wine, which contains on an average 10 per cent. of alcohol, but scarcely 2 per cent. of extractive substances. Fruit-wines, (cider, etc.), with only 5 to 6 per cent. of alcohol but a large quantity of extractive substances, again require different treatment from grape wine, etc., so that, in a certain sense, it may be said there are as many different methods of making vinegar as there are fundamental materials, and by taking into consideration the difference in the chemical composition of the latter, it is evident that there must be just as many varieties of vinegar. Besides acetic acid and a certain amount of water,

every vinegar contains other substances, which, though frequently only present in very minute quantities, nevertheless exert considerable influence upon its properties.

Even vinegar obtained from dilute alcohol shows differences in odor, which depend on the material used in the preparation of the specific alcohol. Potato alcohol always contains traces of potato fusel oil (amyl alcohol), while other fusel oils are found in alcohol prepared from grain or molasses. In the oxidation of the alcohol by the vinegar ferment, these fusel oils are also oxidized and converted into combinations distinguished by their peculiar and very strong odor.

Though these bodies occur in vinegar in such minute quantities that they can scarcely be determined by chemical analysis, an expert can detect them by the sense of smell, and from the specific odor of the vinegar form a conclusive judgment as to the material used in its preparation.

The differences in vinegar from wine, fruit, beer and malt are still more pronounced, and extend not only to the odor, but also to the taste. Besides a specific odoriferous principle, every kind of wine contains œnanthic ether, tartar, tartaric and succinic acids, glycerin, and a series of extractive substances not thoroughly known. The odoriferous substances and the œnanthic ether also undergo alteration in the oxidation of alcohol, and are converted into other odoriferous combinations with such a characteristic odor that wine vinegar can at once be recognized as such by it. On account of the presence of so many substances each possessing a specific taste, that of the wine vinegar must, of course, differ from that of pure dilute acetic acid.

Similar conditions prevail in fruit-wine, beer, malt extract, etc., and hence vinegar prepared from these fluids must possess definite properties.

## CHAPTER VI.

## QUICK PROCESS OF MANUFACTURE OF VINEGAR.

IN 1823 Schützenbach conceived the idea that by greatly enlarging the relative surfaces of contact of the alcoholic solution and air containing oxygen, the process of acetification would be greatly facilitated. His experiments proved successful, and soon after the quick vinegar process was generally adopted. Analogous processes were nearly at the same time invented, in Germany by Wagmann, and in England by Ham.

The principle involved of course depends on an extreme division of the liquid being effected. This is very skilfully contrived. By making the alcoholic solution percolate slowly through, and diffuse over, a mass of shavings, wooden blocks, pieces of coal or cork, etc., it forms a very thin layer, presenting a large surface, and is therefore better adapted for the chemical appropriation of the oxygen in the current of air which is transmitted over it. The mass of shavings, etc., serves not only for the division of the liquid into fine drops, but also as a carrier of the vinegar ferment.

It will be readily understood that this arrangement presents in a high degree all the conditions required for the formation of vinegar, the vinegar ferment upon the shavings acquiring from the liquid all the substances required for its nutriment and propagation, and by the current of air passing through between the shavings is enabled to oxidize the alcohol to acetic acid. This process taking place simultaneously on thousands of points in a normally working generator explains why a large quantity of alcohol can in a comparatively short time be converted into acetic acid. The term quick process is hence very appropriate for this method, it differing from the older slow process only in less time being required for its execution, the chemical processes being the same in both cases.

It will be seen that the generator, technically called "graduator," used in the quick process may be compared to a fur-



nace in which the fuel (in this case the alcoholic fluid) is introduced from above and the air from below. The spaces between the shavings, etc., may be compared to the interstices of a grate, combustion taking place on the points of contact of the alcoholic fluid, vinegar ferment and air. The product of (partial) combustion—the vinegar—collects in a reservoir in the lower part of the generator.

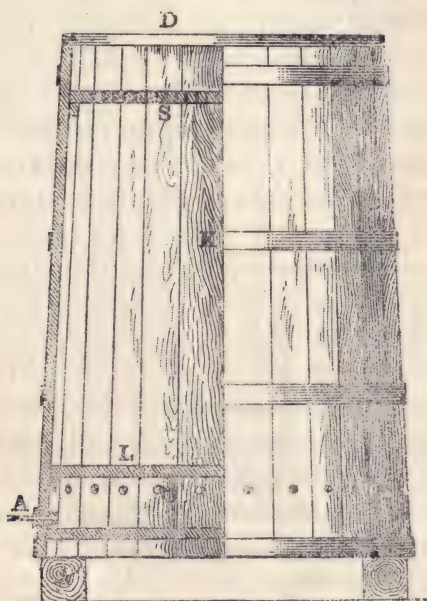
Each generator, as previously stated, requires about 0.4 liter of air per second, which must ascend uniformly from below through the mass of shavings, etc. At the first glance this would seem very simple, but its practical execution is accompanied by many difficulties, and hence a large number of various constructions of generators have been proposed by which this object is claimed to be best attained.

*Generators*—A peculiarly constructed vessel, called the *generator*, is required for the production of vinegar by the quick process. It is divided into three spaces above one another, the uppermost serving for the division of the alcoholic liquid into many small drops; in the center one, which forms the largest part of the apparatus, the alcoholic liquid is converted into vinegar, while the lower one serves for the collection of the vinegar.

The best form of the generator is that of a truncated cone. This form offers to the alcoholic liquid in its passage from the upper part of the generator the opportunity of spreading over a constantly increasing surface, and by thus coming in contact with the fresh air entering the lower part of the apparatus its oxidation must evidently be promoted. The current of air in passing from below to above yields a certain portion of its oxygen in the lower part of the apparatus, and if it were allowed to ascend in a vessel of a purely cylindrical shape, the alcoholic fluid running down would come in contact with air quite poor in oxygen. Hence this evil must be sought to be overcome by the acceleration of the motion of the air upwards, which is accomplished by giving the vessel the form of a slightly truncated cone.

Fig. 3 shows a common form of generator. It consists of the wooden vat *K* provided with a perforated false bottom *L* a few inches from the bottom, and another *S*, similar in structure, at the same distance from the top. The aperture *A* serves for the discharge of the fluid collecting underneath the false bottom *L*. The cover *D*, the arrangement of which will be described later on, serves for regulating the drought of air in the generator. In the lower part of the generator, holes, *O*,

FIG. 3.



are bored. These holes are intended for the entrance of air, and in number may be as many as desired, since the regulation of the current of air is not to be effected on the lower portion of the apparatus, but on the cover.

For the construction of the generator wood thoroughly seasoned and as free as possible from knots should be used. Formerly oak was largely employed for the purpose but, besides its being too expensive, it has the disadvantage of be-

ing so rich in extractive substances that a generator constructed of it, has to be several times lixiviated with water before use, as otherwise the vinegar prepared in it would for a long time acquire a disagreeable tang and dark color. Larch is an excellent wood for the construction of generators. In this country pitch pine is largely used, and is well adapted for the purpose, as it is cheap and readily obtainable everywhere. It is claimed by some manufacturers that the pitch pine protects fermentation in generators constructed of it from the influence of rapid variations in temperature which are of frequent occurrence in portions of this country.

The hoops of the generators, as well as all other metallic parts in the factory, should be coated with good linseed-oil varnish or asphaltum lacquer, and care should be had immediately to repair any injury to this coating, as otherwise heavy rusting is caused by the vapors of acetic acid contained in the air of the work room.

There is considerable variation in the dimensions of the generators, some having only a height of 5 feet, with a lower diameter of 3 feet 3 inches, and others again a height of 20 feet or more, with a diameter of up to  $6\frac{1}{2}$  feet. The small generators have the disadvantage of rapidly yielding heat to the exterior, and hence a correspondingly high temperature must be maintained in the workroom in order to keep up the proper degree of heat in their interior. On the other hand, generators of considerable height have the drawback of the shavings, etc., with which the center space is filled, becoming strongly compressed by their own weight, thus obstructing the proper passage of the air. It has been sought to overcome this evil by placing several false perforated bottoms in the generator, in order to divide the weight of the filling into as many smaller weights as there are false bottoms. But this arrangement is also attended with inconveniences, it being difficult to maintain a sufficiently strong draught of air in generators of such height.

Some manufacturers hold that the production of very



strong vinegar containing 11 to 12 per cent. of acetic acid is only possible in very tall generators. This opinion is, however, unfounded, the manufacture of very strong vinegar being just as well or rather better effected in small generators than in those twenty feet or more high, which besides are very expensive.

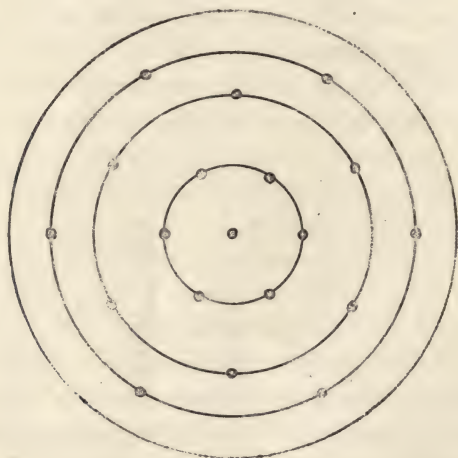
The manufacture of vinegar should be carried on in a room with a low ceiling, since even with the best heating arrangement the temperature near the ceiling is always much higher than on the floor. However, with the use of generators 20 feet high, the ceiling of the work room must be at least 26 feet high, which makes it impossible to maintain a uniform temperature, as the difference between the upper and lower parts would frequently amount to more than  $25^{\circ}$ .

The most suitable generators are very likely those with a height not exceeding 10 feet, and a lower diameter of about 45 inches and an upper one of about 35 inches. A large diameter, to be sure, contributes towards the maintenance of a uniform temperature in the generator, but it has the disadvantage of making it difficult for the air to ascend uniformly through all parts of the filling. This drawback is sought to be overcome by placing in the center of the generator a tube open above and below and provided on the sides with holes. Such tube, however, does not produce the intended favorable effect upon the draught of air in the parts of the filling surrounding it, experience having shown that the greater portion of the warm current of air ascending in the interior takes the nearest road to the top, *i. e.*, through the tube, without passing sideways into the filling. Every generator of suitable construction should be provided with a well-fitting cover. In this cover, Fig. 4, are bored, in concentric circles, holes which are intended for draught apertures. If the draught of air in the interior is too great, it can be at once diminished by closing a number of these holes, it being even possible to direct it towards a certain portion of the filling. This arrangement is, however, only available when the false bottom to be described later on is

either not used or provided with a number of short vertical tubes which permit the passage of the air.

Many generators are provided with a number of obliquely bored apertures below the false bottom through which the air can escape. This is, however, attended with the disadvantage that a regular draught of air only takes place in the outer layers of filling next to the walls, while it is not sufficiently strong in the center of the apparatus. It is also incorrect to have but one air aperture in the cover, which can be made larger or smaller by means of a slide. In a generator thus arranged, the

FIG. 4.



current of air entering below will naturally pass chiefly through the conical portion of the filling, the base of which is formed by the lower false bottom and the apex by the draught aperture in the cover. The lower portion of the filling, which embraces this cone, remains without sufficient ventilation and is ineffective as regards the oxidation of alcohol.

In Figs. 5 and 6 the hatched surfaces terminated by the dotted lines illustrate the portions of the generator in which, with the use of many apertures below the false bottom and a single one in the center of the cover, the regular current of air

from below to above passes. Although a current of air takes place outside of these lines, it is in most cases too weak, and consequently the entire available space of the generator is not sufficiently utilized.

FIG. 5.

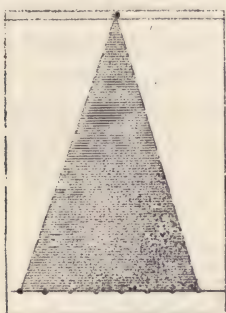
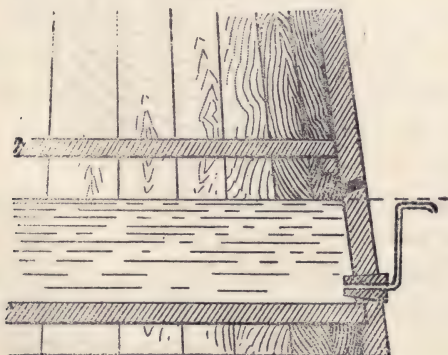


FIG. 6.



Each generator may also be entirely open below and stand in a shallow tub, which serves for the collection of the vinegar. Generally, however, the lower portion of the generator itself is used for this purpose, and is provided with an arrangement for

FIG. 7.



the occasional discharge of the collected fluid. This can be effected either by a spigot fixed immediately above the bottom or, as in Fig. 7, by a glass tube, which bends upwards nearly as high as the air-holes and then curves downward so as to dis-



charge the liquid, when it rises as high as the shelf in the interior of the apparatus, into an appropriate vessel placed to receive it. Simple as this arrangement is, it is scarcely suitable in the practice on account of its being too liable to breakage, and hence it is better to provide the generator with an ordinary spigot, and prevent the vinegar from rising too high, by boring about  $\frac{1}{2}$  inch below the draught apertures a hole in which is fitted a pipe leading to a tub. The vinegar rising to the height of this pipe will commence to run off, and thus give warning to empty the generator by opening the spigot.

FIG. 8.

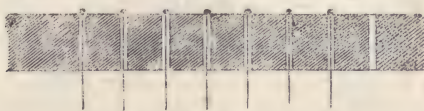


In generators of older construction a strong hoop is fixed about one foot from the top, on which is placed a perforated disk which serves for distributing the alcoholic fluid as uniformly as possible over the entire filling. The disk, Fig. 8; is perforated with numerous holes (about 400 with a disk diameter of 3 feet) arranged in concentric circles. These holes are loosely filled with cotton wick or packthread, a knot being made at the top end to keep them from falling through. The threads reach down to the shavings, and serve the double purpose of conducting the liquid equally through the body of

the generator and also of stopping it from passing too rapidly through it (see Fig. 9). It is important to pack the disk so tightly against the walls of the generator that none of the liquid can percolate, which is best effected by a packing of tow, and coating this with a mixture of equal parts of wax and rosin. The dripping of the alcoholic fluid through the disk taking place uniformly only when the latter lies perfectly horizontal, great care must be exercised in placing the generator. To prevent warping several strong cross-pieces are inserted in the lower side of the disk.

As previously mentioned the current of air must pass through

FIG. 9.



all portions of the filling, and for this purpose seven short glass tubes, *r* (Fig. 8), about  $\frac{3}{4}$  inch in diameter, are inserted in the disk. These tubes are so arranged that one is in the center of the disk and the others in a circle equidistant from the center and the periphery. Upon the disk is placed the well-fitting cover, provided with an aperture for the passage of the air. This aperture, about 3 inches square, is provided with a well-fitting slide, so that it can be made larger or smaller at will. As previously stated, it is more suitable to provide the cover with a large number of draught holes arranged in concentric circles and to fit each hole with a wooden stopper. By withdrawing or inserting the stoppers the draught of air can then be properly regulated.

To effect the influx of air from below in such a manner that it takes place not only through the draught holes in the circumference, but also assures its conveyance to the center of the apparatus, it is recommended to insert in the center of the lower part in which the fluid collects a tube, *R*, Fig. 10, open

at both ends and protected above by the hood *H* against the dropping in of alcoholic liquid.

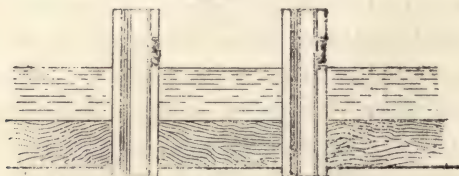
A uniform distribution of the alcoholic liquid upon all portions of the filling of the apparatus would be effected if about

FIG. 10.



the same quantity of liquid dripped from all the threads. This being, however, difficult to attain, it has been sought to give the disk a more suitable arrangement, which consists, for instance, in the insertion of small wooden tubes with a small

FIG. 11.



aperture on the side (Fig. 11). This arrangement, though very suitable in itself, becomes, however, useless in case of the slightest warping of the disk, a number of the tubes being then raised so high that no fluid runs through them, while it passes in a full stream through the others.



These drawbacks connected with the use of a disk can be somewhat diminished by the employment of a so-called "tilting trough" (Figs. 12 and 13), which is arranged as follows:—

Upon a perfectly horizontal axis is placed a rotatory, trough-like vessel divided by a partition into two equal parts.

If the tilting trough is in the position shown in Fig. 12, the alcoholic liquid runs through the cock, placed above, into the partition marked 1.

As soon as this partition is filled to a certain height it turns over in consequence of the disturbance of the equilibrium of the trough and assumes the position shown in Fig. 13. In this position partition 2 is gradually filled with alcoholic liquid; the trough then tilts back into position 1, and so on.

FIG. 12.

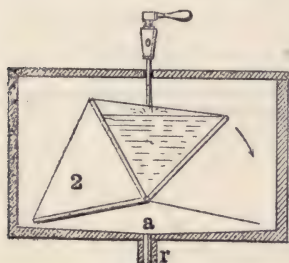
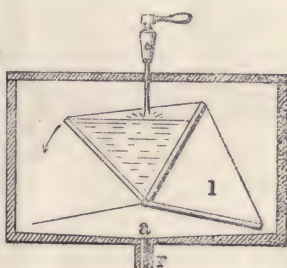


FIG. 13.

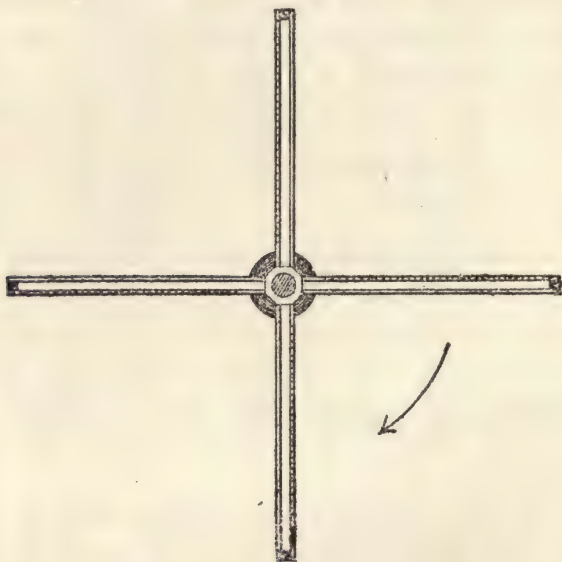


It will be seen that with the assistance of such a tilting trough the same quantities of liquid can always be poured out at certain intervals, and that this arrangement can be used for distributing the alcoholic liquid upon the disk, the latter in this case being best provided with holes having the form of an inverted cone. The apex of this cone forms a very narrow aperture through which the alcoholic liquid poured upon the disk trickles in very thin jets upon the filling of the generator.

But even this arrangement is not free from objections, it working entirely satisfactorily only as long as the disk remains in a perfectly horizontal position. In the more modern constructions of vinegar generators the disk is generally entirely

omitted and the distribution of the alcoholic liquor effected by a so-called "sparger," similar to the one used in beer brewing for sprinkling malt residues. The sparger is arranged like a simple turbine, and is moved by reaction in the direction opposite to that in which the discharge of the fluid takes place. Spargers used in vinegar factories can be constructed only of a material indifferent to the action of acetic acid, such as wood, glass, hard rubber, etc. Their construction will be understood

FIG. 14.

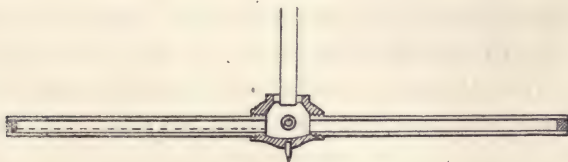


from Figs. 14 and 15, showing a view from above and a cross-section.

Into a hollow cylinder of wood are screwed four thin wooden tubes, closed at both ends and perforated lengthwise with numerous small holes. The tubes are so arranged that all the holes are directed toward one side. The basin in the center is closed on top by a glass tube about 20 inches long and of sufficient width to allow of the passage of as much fluid as can at one time run off through all the lateral tubes.

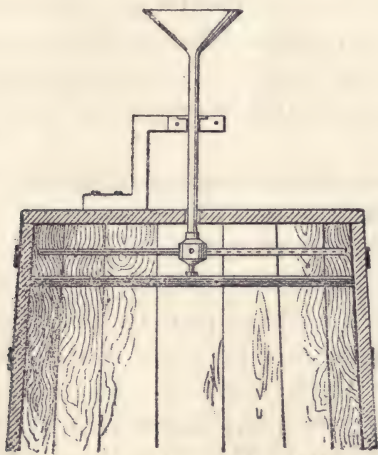
The principal requisite of the correct working of the sparger is that it revolves with ease around its vertical axis. This is effected by placing in the center of the vessel a glass pin drawn out to a fine point and running in a small glass step. The

FIG. 15.



vertical glass tube is guided in a sharp-edged wooden ring fastened to a stay placed upon the cover of the generator (Fig. 16). The sparger finds its center of motion upon a strip inserted in the direction of the diameter of the generator. This strip is placed at such a height that the sparger can move

FIG. 16.



freely between it and the cover of the generator. The sparger being in position as shown in Fig. 16, a funnel-shaped vessel, through which the alcoholic fluid is poured in, is placed upon the glass tube.



By now pouring through this funnel-shaped vessel the alcoholic liquid in a sufficiently strong stream, so that during its influx the glass tube remains filled, it passes in fine jets through the lateral openings, and, the sparger revolving in an opposite direction, is distributed in the form of a fine spray over the filling in the generator.

The use of the sparger overcomes the difficulties frequently occurring with the disk, especially as regards the position of the latter, and the circulation of air through the apparatus also takes place in a perfectly uniform manner. A number of apertures in the cover of the generator serve also here for the regulation of the current of air.

A thermometer is an indispensable adjunct to a generator, and should be so placed that the temperature prevailing in the apparatus, and especially in the center, can be readily read off. This is best effected by introducing at about half the height of the apparatus, through an obliquely bored hole in one of the staves, a glass tube closed at the lower end and reaching to the center of the filling. This tube serves for the reception of a thermometer fastened to the lower end of a stick of wood. The latter projects from the glass tube, so that the thermometer can be quickly drawn out and the temperature read off.

*Filling the generators.*—The space between the upper disk and lower false bottom is filled with a material offering a large surface for the distribution of the alcoholic liquid. Pieces of charcoal and of pumice washed in hydrochloric acid and well rinsed in water to remove empyreumatic substances, which would render induction of acetic fermentation impossible, have been used for the purpose. Small pieces of cork and cork waste have also been recommended for filling. This material absorbs liquids like a sponge, but when sucked full does not press evenly together, dry places being thus formed during the operation. Corn cobs thoroughly dried and finely divided may be used to advantage, especially in the manufacture of wine and cider vinegar. Grape stems are still occa-

sionally used. They actually present a very large surface but, independent of the fact that they cannot be everywhere obtained in sufficient quantities, they have the drawback of becoming in a short time so firmly compressed as to prevent the free passage of air.

Beechwood shavings, however, are now almost generally employed for filling the generators. Indeed, beechwood presents many advantages: It can be had easily and is cheap; it curls well and stands without breaking for a length of time. White woods will curl as well, but they will not stand so well as beech; resinous woods are not porous enough, and besides their rosin is objectionable, as it may partly dissolve in the vinegar; oak wood does not curl as well and contains too much coloring matter and tannin.

The beech shavings are generally made in special factories. They consist of wooden bands about 0.02 inch thick,  $1\frac{1}{2}$  inches wide, and 16 to 20 inches long. They are rolled into close spirals by a special machine, and each shaving, according to the above dimensions, presents a surface of about 62 square inches. Now, as a generator of moderate size contains many thousands of such shavings, it will be readily seen that the surface over which the alcoholic fluid is distributed is an extraordinarily large one.

A shaving of the stated dimensions represents in a rolled state a cylinder with a volume in round numbers of 1.7 cubic inches. By allowing an interspace of .85 cubic inch between the shavings,  $1.7 + 0.85 = 1.92$  cubic inches space is required for each shaving. The space to be filled with shavings in a generator 3.28 feet in diameter and 6.56 feet high is equal to 55.44 cubic feet, and hence 58,000 shavings, with a total surface of 22,733.56 square feet, are required for the purpose. Now suppose only 5 per cent. of this surface is continually active in the formation of vinegar, we have still a surface of over 1075 square feet at our disposal. But the active surface would seem to be actually much smaller even with the most favorable working of the generator, as otherwise the average

quantity of alcohol daily converted into acetic acid in a generator would be much larger than is actually the case.

Beechwood shavings contain a considerable quantity of extractive substances, which if not removed, would for a long time impart a disagreeable tang (woody taste) to the vinegar. Hence it is recommended to lixiviate the shavings in water repeatedly renewed, in order to get rid of the substances soluble in cold water, and remove the last traces of them by treatment with steam.

This steaming is best effected in a large tub or vat, which is later on to be used as a generator. The shavings are thrown in loosely and covered with a loaded lid. A steam-pipe is introduced through a hole near the lid, and the tap-hole near the bottom is opened. The steam-pipe being connected with a boiler, in which prevails a tension of  $1\frac{1}{2}$  to 2 atmospheres, the steam-cock is at first opened but slightly, to prevent the steam entering with great force from throwing off the lid, or even bursting the vessel. In the commencement of the operation the steam condenses on the shavings, but after some time the vessel becomes very hot, and a dark-colored fluid, consisting of almost boiling water charged with extractive substances of the wood, begins to run off. After continuous steaming for about 20 to 60 minutes—according to the size of the vessel—the fluid running off becomes clearer until finally clear water is discharged, which is indicative of the removal of the extractive substances soluble in water.

Although not absolutely necessary, it is advisable to dry the steamed shavings. When air-dry they still contain about 20 per cent. of water, which in the subsequent "acetification" of the generator must be replaced by vinegar. Hence it is recommended to dry the shavings completely by exposing them for some time to a current of air of  $194^{\circ}$  to  $212^{\circ}$  F.

In a factory provided with a central heating apparatus\* in the cellar, this drying of the shavings can be effected without

\* The arrangement of a central heating apparatus will be described later on in speaking of the arrangement of the factory.



difficulty, it only being necessary to put them in a vessel with a perforated bottom and open on top, and place the vessel over an aperture of the register through which the hot air from the heating apparatus ascends, closing all other apertures.

As perfectly dry wood absorbs with avidity moisture from the atmosphere, the shavings thus dried should immediately be brought into another vessel and, while still hot, moistened with the vinegar intended for acetification.

Before using the shavings for filling the generators, it is necessary to allow them to swell by placing them in water or alcoholic liquid. If this were omitted and the shavings introduced in a dry state, they would rise above the generators as soon as moistened, on account of the increase in volume by swelling.

In most factories it is customary simply to pour the shavings into the generator, but for a uniform distribution of the alcoholic fluid it is advisable to proceed with the filling in a certain order. First place the shavings in three or four regular layers upon the false bottom, then pour them in loosely to a height of 8 to 12 inches, and after leveling the surface as much as possible pour in again, and continue in this manner until the generator is filled. The uppermost portion should again consist of three or four regular layers.

All the generators used in a vinegar factory should be of the same size and charged with the same number of shavings, which is best effected by filling them with the same quantity by weight. The total surface of shavings being thus nearly the same in all generators, the latter will work uniformly, *i. e.*, with an equal temperature and draught of air; and in the same time convert equally large quantities of alcohol into acetic acid.

## CHAPTER VII.

## ARRANGEMENT OF A VINEGAR FACTORY.

THE arrangement of the manufacturing rooms formerly customary even in large factories is by no means a suitable one. The generators were generally simply placed in a room adapted for the purpose by its size, while the high temperature required was sought to be maintained by heating. By considering, however, that every considerable variation in the temperature causes also a disturbance in the formation of vinegar, it will be seen that the object of keeping up an undisturbed working of the factory cannot be attained by such primitive means. A suitable arrangement of the room in which the vinegar is to be manufactured is, therefore, absolutely necessary.

The principal requisites to be observed are: The maintenance of a uniform temperature in the room and a suitable arrangement for ventilation. Further, simple devices for the conveyance of the raw materials and the finished product must be provided for, and means devised for regaining the acetic acid, with the vapors of which the air in the manufacturing room is constantly saturated.

For the maintenance of a uniform temperature in the work-room, which should remain almost constant even in the coldest season of the year and during abrupt changes in the outer temperature, the walls should be of more than ordinary thickness and the number of windows and doors sufficient only for the necessary light and communication, and so arranged that no unintentional ventilation can occur. The windows and doors should, therefore, be double, and the latter so placed that one can be closed without opening the other. The walls and ceilings should be plastered and preferably papered with heavy packing paper; asbestos shingles are also highly recommended for this purpose.

Asphaltum being impermeable and also indifferent to the action of acetic acid, is undoubtedly the best material for the floor of the workroom, though it may also be constructed of large slabs of sandstone with the joints filled in with asphaltum. Cement floors can only be recommended provided they are immediately after their construction coated with silicate of soda until they cease to absorb it. In constructing the floor care must be had to give it such an inclination that the entire surface can be cleansed by a simple jet of water. If the heating channel is conducted lengthwise through the workroom, gutters for the rinsing water to run off must be arranged on both sides.

The height of the room depends on that of the generators.

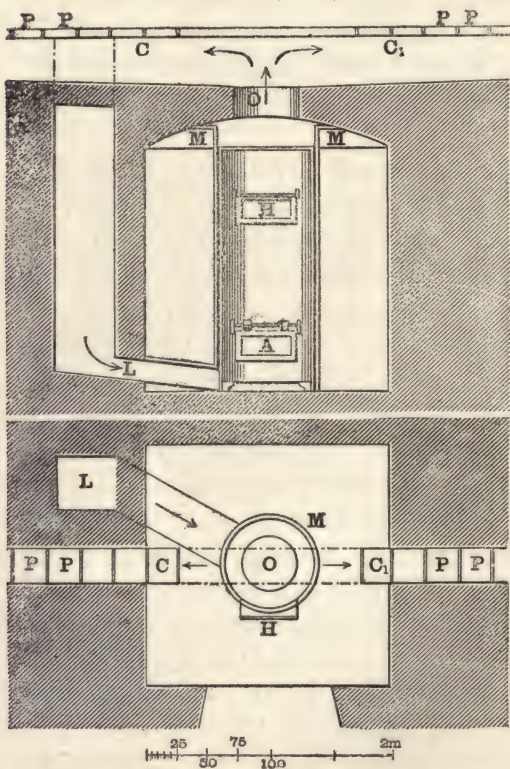
*Heating of the Workroom*—Heating by a stove placed in the workroom itself can only be recommended for very small factories; in larger ones a special heating apparatus should always be provided. Where stoves are used it is recommended to arrange them so that the fuel can be supplied and the ashes removed from the outside, *i. e.*, from a room adjoining the actual workroom. In attending to the stoves fine particles of ashes will unavoidably reach the air, and from the latter they may get into the generators, and being soluble in acetic acid may injure the vinegar ferment.

For large factories a heating apparatus similar to the one shown in Figs. 17 and 18 can be recommended. The heater, provided with the feeding-door *H* and the air-regulating door *A*, stands in a vault beneath the center of the room to be heated. It is surrounded on all sides by the sheet-iron jacket *M*, reaching from the floor of the cellar to the top of the vault. In the vault is a circular aperture, *O*, for the reception of the flues *C* and *C*<sub>1</sub>. The latter ascending slightly, run along the center of the room to be heated. Above they are covered by cast-iron plates, *P*, and by pushing these plates apart or substituting a lattice plate for one of them in any part of the flue, warm air can be admitted to the room. If the room is to be heated without renewing the air, the register in the flue *L*,



which communicates by a flat iron pipe with the lower part of the jacket, is opened. The furnace being heated, the air in the room is sucked in the direction of the arrow through the flue *L*, and passing between the jacket and the furnace, ascends strongly heated through *O* and penetrates through the openings in the flue, air being again sucked through *L*, and so on.

FIGS. 17, 18.



If, however, the air in the workroom is to be entirely renewed, the air-flue *L* is closed and a register (not shown in the illustration) in the lower part of the jacket opened. In this case the air in the cellar is sucked in, heated and distributed through the flues *C* and *C*<sub>1</sub>. By partially opening this

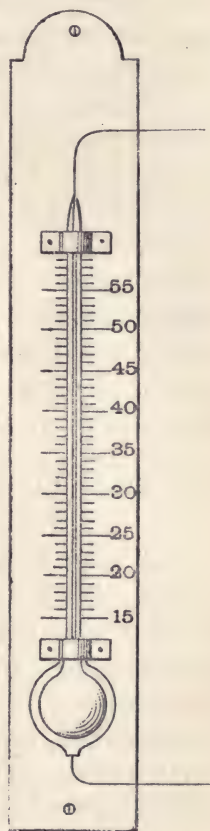
register and that in *L*, a portion of the air can be renewed at will.

In order to be able to form a correct idea of the state of the temperature prevailing in the room, it is advisable to have several ordinary thermometers and also a maximum and minimum thermometer. If the latter shows no greater variation than from  $4^{\circ}$  to  $5^{\circ}$ , the process of heating may be considered as satisfactory.

A very suitable apparatus for controlling the temperature in a vinegar factory is an electrical thermometer, which is so arranged that a bell rings in case the temperature rises above or falls below a certain degree. By placing two such thermometers in the room, the bell of the one indicates the rise of the temperature above the limit, and that of the other that it has fallen below it.

Fig. 19 illustrates the principle of a maximum electrical thermometer, *i. e.*, one which rings a bell when the temperature of the room exceeds a certain limit. Into the bulb of an ordinary mercury thermometer is melted a platinum wire; another platinum wire is inserted in the tube up to the mark indicating the temperature not to be exceeded, for instance,  $35^{\circ}$  C. The ends of the platinum wires projecting from the thermometer are connected by insulated copper wires with a galvanic battery consisting of several elements, an ordinary door-bell being inserted in one part of the conductor. If now, in consequence of a continued increase in the temperature, the mercury rises to the point of the platinum wire at the figure  $35^{\circ}$ , the circuit of the battery is closed at the same time by the column of mercury, and the bell rings and keeps ringing until the circuit is again opened by the mercury falling below  $35^{\circ}$ .

FIG. 19.



The minimum electrical thermometer, used for indicating the falling of the temperature below a certain degree, is so arranged that one platinum wire is melted into the bulb of the thermometer and the other in the tube at the point below which the temperature is not to fall. As long as the mercury remains above this point a battery, which changes a piece of iron to an electro-magnet, whose armature opens a second battery which is connected with an electric bell, remains closed. If the temperature falls below the minimum, the circuit of the first battery is opened, and the armature of the electro-magnet falling down effects the closing of the second battery and sets the bell ringing.

By placing such thermometers not only in the working room but also in every generator, the control of the entire process would be immensely facilitated, but at the present time these useful and at the same time inexpensive instruments are but little used in vinegar factories.

In factories arranged according to the automatic system, the alcoholic liquid is contained in vessels placed at such a level that their contents can run directly into the generators. The alcoholic liquid having to be correspondingly heated, adequate provision must be made for heating the space in which the reservoirs are placed. In order not to increase the height of the entire room, it is recommended to place these vessels in the center and give only to this portion the required height. This has the further advantage that the alcoholic liquid can be pumped up by the use of a pump with a short rising-pipe, and the liquid can be readily conducted from the reservoirs to the separate generators by means of pipes.



## CHAPTER VIII.

## ARTIFICIAL VENTILATION OF THE VINEGAR GENERATORS.

THE first experiments in conveying direct air to every generator were made in England; but though this step towards improvement in making vinegar must be considered an important advance, the English process failed of being accepted in practice on account of the inadequacy of the apparatus used.

In the English factories by a special apparatus a current of air was sucked from above to below through every generator. As shown in Fig. 20, the tall generator is open on top and divided into several partitions by false bottoms, upon which the shavings, etc., rest. Above each false bottom holes are bored in the circumference of the generator. In the bottom of the generator is inserted a pipe which is connected with an arrangement for sucking in the air, a blower or air-pump being used for the purpose.

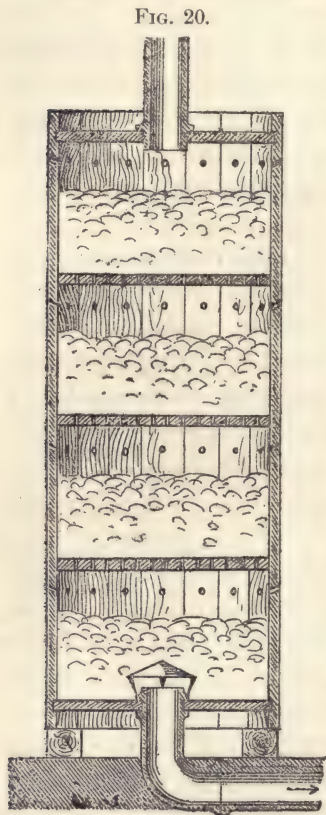
As will be seen from the illustration, the suction of air through all parts of the generator cannot be uniformly effected by the use of this apparatus, the current of air being much more checked in the upper portions by the false bottoms and holes in the circumference, than in the lower. Hence the effect of the air-pump or blower will chiefly assert itself in the lowest partition. This drawback might be remedied by leaving out the false bottoms and placing no air-holes in the circumference of the generator entirely open at the top. By this means the air would be forced to pass in a uniform current through the entire layer of the filling material.

That the passage of the current of air from above to below is entirely incorrect, because contrary to all theoretical requirements, can readily be explained: In a generator in full activity, oxidation of alcohol must already take place in the uppermost portion, and hence a certain quantity of oxygen is withdrawn

from the air. This process being also continued in the lower parts of the generator, a current of air already deprived of a portion of its oxygen, and hence less suitable for the further

formation of acetic acid, would be sucked in the same direction which the drops of alcohol take.

The principal reason advanced for the use of a current of air from above to below is that by these means a uniform temperature is maintained in all parts of the generators, while it rises considerably in the upper part of those in which the air passes from below to above. This rise of temperature is, however, agreeable to nature. The air entering from below oxidizes the alcohol to acetic acid, becoming thereby poorer in oxygen and again heated. By the higher temperature it acquires, it is, however, capable of a more vigorous chemical activity, so that it will induce the process of the formation of vinegar, even in the uppermost portions of the generator. Besides, the warmer current of air moving upwards has the further advantage of yielding heat to



the drops of alcoholic fluid trickling down. With the use of generators of moderate height, and with a suitable regulation of the draught of air, the maximum temperature will not be exceeded, even in the uppermost portions of the generator.

If no rise of temperature is observed in the lower portions of a generator in which the air passes from above to below, it only proves that the air has lost too much oxygen to further effect a vigorous oxidation of the alcohol. It will be readily

understood that under these conditions a diminution in the loss of substance can, to a certain degree, be effected, but it is doubtful whether the generators are utilized in the manner they should be; besides, the diminution in loss of substance cannot be very considerable. Since a high temperature also prevails in ventilated generators, the current of air passing downward will be loaded with as much vapor of alcohol or of acetic acid as it can absorb at this temperature, and, hence, it would seem, no diminution in loss by evaporation could be effected. To render this possible, the current of air sucked from the generator would have to be sufficiently cooled off by a suitable arrangement to allow of the greater portion of the vapors carried away by the current of air being condensed to a fluid.

*Schulze's Ventilating Apparatus.*—The ventilation of the vinegar generators, according to the previously described method, requires the presence of an uninterruptedly acting power for working the air-pump, blower, etc. As is well known, a current of air can, however, be also produced by heating the air passing through an ascending pipe, by which it becomes specifically lighter and ascends, while denser air enters from below, etc. Schulze, as will be seen from Fig. 21, has applied this method to the ventilation of vinegar generators.

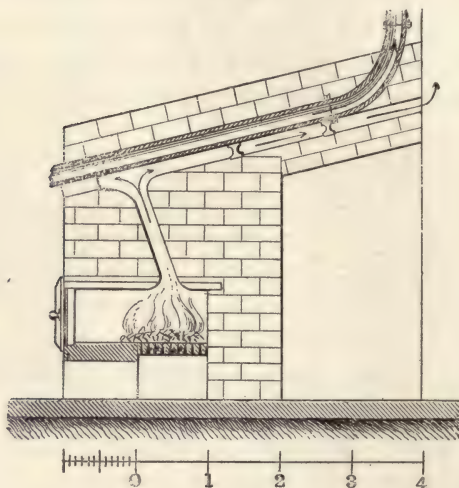
Schulze's generator differs somewhat from the ordinary construction, and is arranged as follows: The vat has a height of about 8 feet, and a diameter of 2 feet 6 inches. In the upper part it is terminated by a false head, fitting air-tight, and is further provided with a cover, in the center of which is an aperture about  $2\frac{1}{2}$  inches in diameter, which serves for the entrance of air, while another aperture on the side serves for pouring in the alcoholic liquid. In the false head are inserted four glass tubes, open at both ends, and about  $\frac{3}{4}$  inch in diameter, which afford a passage to the air. The generator is filled with pieces of washed and assorted charcoal, so that pieces of the size of a nut are placed upon the false bottom, and upon this smaller pieces, gradually decreasing in size



until those on the top are only that of a pea. In the center of the bottom is inserted a wooden tube, open at both ends and provided on top with a hood to prevent the trickling in of vinegar (see Fig. 10). By a suitable intermediate piece, this tube is connected with the draught-pipe (see Fig. 21), in which the ascension of the air by heating is effected.

The draught-pipes are of cast-iron, and are about  $\frac{1}{2}$  inch thick and about  $4\frac{1}{2}$  feet long, with a clear diameter of 2 inches.

FIG. 21.



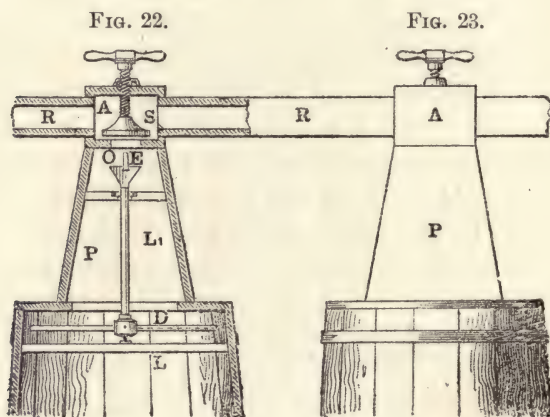
They are placed, strongly inclined, over the flues of a heating apparatus and covered above by a double course of stone. The air in the iron draught-pipes, being heated by the escaping gases of combustion, ascends and effects the passage of a current of air from above to below in the generators. For keeping up a constant ventilation it is claimed to be sufficient to heat the furnace only once a day. With this construction it is necessary to have as many draught-pipes as there are generators. The same effect might, however, also be attained by connecting the pipes leading from several generators with a draught-pipe of a somewhat greater diameter and length.

It is not difficult to prove that a uniform ventilation of the generators cannot be obtained by the use of this construction. As long as the draught-pipes are strongly heated, a very rapid current of air will pass through them and the generators connected with them, which will, however, decrease in the same degree as the pipes cool off. Hence, in the first case, a too rapid current of air, accompanied by a correspondingly strong evaporation of alcohol, would pass through the generators, and in the latter, ventilation would be so sluggish that the process of the formation of vinegar would not proceed in a normal manner.

*Generators with Constant Ventilation and Condensation.*—The object to be attained by the use of special ventilating contrivances is a double one: To conduct a constant current of air through the generators, and, further, not to allow the temperature to rise above a certain limit, so as to decrease by these means the loss by evaporation of alcohol and acetic acid. This object can, however, be attained only by the use of an apparatus which allows of the most accurate regulation of the current of air passing through the generator, and is connected with a contrivance by which the vapors of alcohol and acetic acid carried along by the current of air can be condensed as much as possible. The apparatus described below is well adapted for the purpose. Its principal parts consist of the generator, the apparatus for condensing the vapors, and the ventilator.

The construction of the lower part of the generator, Fig. 22, is the same as of those previously described. The cover fits tightly upon the upper edge of the vat, the joint being made air-tight by strips of paper pasted over it. In the center of the cover is a square aperture, from which rises a quadrangular pyramid, *P*, constructed of boards, upon which sits a low prism, *A*. The sparger *D* has its center of motion upon the strip *L*, placed in the uppermost portion of the generator, and is guided above in the short strip *L*<sub>1</sub>, which carries the sharp-edged ring described on page 51. *E* is the glass tube through

which the alcoholic liquid flows into the funnel of the sparger. On the point where the pyramid passes into the prism *A*, is a bottom provided with a circular aperture, *O*,  $2\frac{1}{2}$  to 3 inches in diameter. Upon the top of the prism *A* is placed a nut, in which runs a wooden screw, provided on the lower end with a wooden disk, *S*, of a somewhat greater diameter than the aperture *O*. By raising or lowering this screw, the aperture *O* can be closed more or less or entirely, and thus the strength of the current of air regulated at will in every generator. The prisms *A* of all the generators are connected with each other by the conduit *R*, constructed of boards.



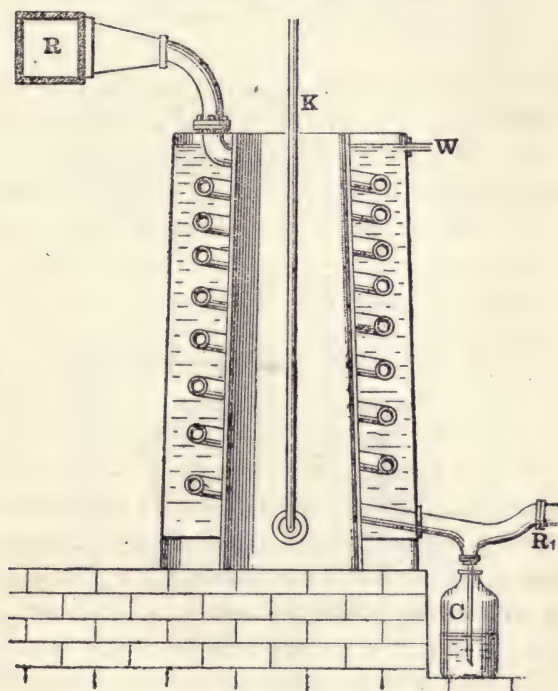
This conduit *R* is connected—best in the center between an equally large number of generators—with the condensing apparatus, the chief feature of which is a worm similar to that used in a still. Fig. 24 shows the apparatus in cross-section.

In a sheet-iron vessel of the same height as the generator is placed another vessel, so that there is a distance of about  $5\frac{1}{4}$  inches between the walls. From a reservoir situated at a higher level cold water runs into the apparatus through the pipe *K*, and off through the short pipe *W*. In the space between the walls of the two vessels lies a tin coil with very thin



walls and a diameter of at least  $2\frac{1}{8}$  inches. On top this tin coil is connected with the wooden tube *R* (Fig. 23) and below with the iron pipe *R*<sub>1</sub>, which leads to the ventilating apparatus. *C* is a glass tube about 16 inches long and  $\frac{1}{2}$  to  $\frac{3}{4}$  inch

FIG. 24.



in diameter, which reaches nearly to the bottom of the flask half filled with water.

The ventilating apparatus consists of an ordinary self-feeding stove, but its jacket is closed below so that air can only pass in between the heating cylinder and the jacket through the pipe *R*<sub>1</sub> coming from the condensing apparatus.

The apparatus works as follows: According as combustion in the stove proceeds slowly or quickly by the corresponding position of the regulating register, the air between the heating

cylinder and the jacket becomes less or more heated and ascends with corresponding velocity. But as the further entrance of air can take place only through the pipe  $R_1$ , the tin coil, and the wooden tube  $R$ , a uniform current of air from below to above must pass through all the generators. To regulate the strength of the current for each generator, it is only necessary to close the aperture  $O$  (Fig. 22) more or less by raising or lowering the screw.

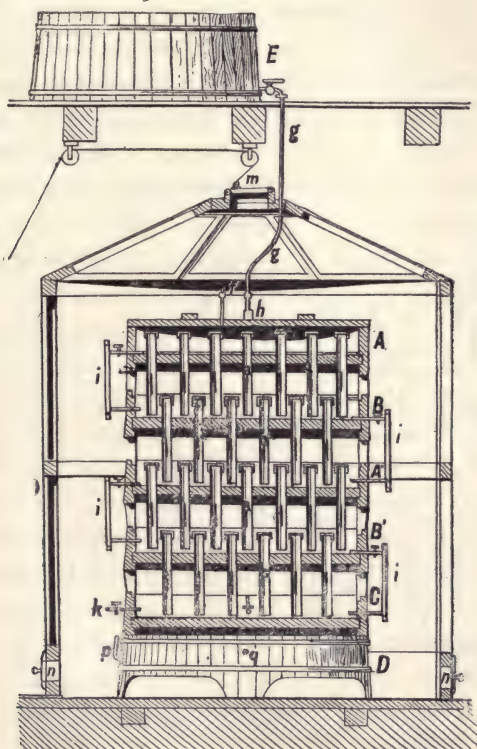
The current of air passing from the wooden tube  $R$  into the tin coil carries with it the total amount of evaporated alcohol or acetic acid. By passing through the tin coil, which is cooled by the water, the air itself becomes cooled off, and the greater portion of the vapors held by it condense to liquid and run off through the tube  $C$  into the bottle. The fluid thus obtained consists chiefly of alcohol, water, and acetic acid, and is again used for the preparation of alcoholic liquid. On account of the peculiar form of the cooling vessel but little water is required for feeding it. As the quantity of vapor separated from the air will, however, be the greater the more energetically the tin coil is cooled off, it is recommended to reduce the temperature of the water to nearly  $32^{\circ}$  F. by throwing in pieces of ice.

It has been proposed to regain the vapors by conducting the air containing them into a large vessel in which water in the form of a fine spray trickles down or is injected. It is, of course, possible in this manner to condense the greater portion of vapors of a higher temperature and tension, but with vapors of at the utmost  $95^{\circ}$  F. little success would be attained. The greater portion of the vapors remaining uncondensed, a very large quantity of fluid containing but little alcohol would be obtained in the course of a day, and this fluid could at the best be used only instead of water for the preparation of alcoholic liquid. The value of the material thus regained would not cover the working expenses of the apparatus. By working, however, with the condensing apparatus described above, the condensed alcohol does not even contain the total quantity of water evaporated with it, and it need only be compounded

with the corresponding quantity of water and vinegar again to yield alcoholic liquid.

The generator manufactured by Singer, of Berlin, shows an essential difference in construction from those previously described. It consists of several shallow wooden vessels arranged one above the other and connected with each other by wooden

FIG. 25.



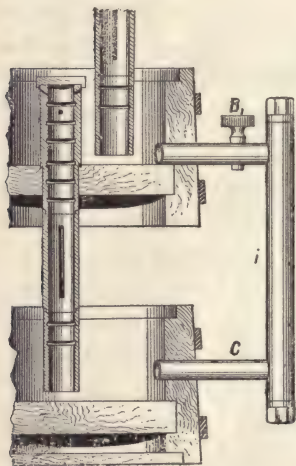
tubes so that the alcoholic fluid runs drop by drop from one vessel into the other, passing thereby through the tubes. In order to distribute the fluid as much as possible, the tubes are inside provided with horizontal gutters, whereby the surface of the fluid passing through is greatly increased. In addition,



each of the tubes is provided in the center with a slit running length-wise, allowing of a free passage of the air. The latter encounters in the tubes the finely-divided fluid and effects the oxidation of the alcohol to vinegar. This is repeated four times and oftener before the alcoholic fluid has passed through the apparatus, whereby, it is claimed, a very complete formation of vinegar is attained. The entire apparatus stands in a case which protects it from cooling off and from too great an access of air, and which can be heated in winter.

Fig. 25 shows Singer's generator in cross section, and Fig. 26 the separate vessels and their connection by drip tubes. Fig. 25 represents five shallow vats standing one above the other, at uniform distances, the latter being effected by elongated staves.

FIG. 26.



In the bottoms of the vats *A* and *A*<sup>1</sup> are inserted 37 tubes, *a*, *b*, by which they are connected with the vats *B* and *B*<sup>1</sup> below. The bottoms of the latter are provided only with 32 tubes, which above connect the vat *B* with *A*<sup>1</sup> and below pass into the vat *C*.

As will be seen from Fig. 26, the sections of the tubes through which the alcoholic fluid is to run slowly are provided above with six annular gutters. Above these gutters are four apertures for the entrance of the alcoholic fluid. In the center the tubes are slit lengthwise for the free admission of air. The lower end of each tube is also provided with two gutters. On top each tube is closed with a lid, but the end entering the vat below is open.

Of all the five vats only the upper one, *A*, is provided with a cover, and upon the latter is fixed a holder, *f*, for securing the tube *g*. Above, the latter is connected with the reservoir

*E*, filled with alcoholic fluid and below ends in the short pipe *h*, which permits the alcoholic fluid to run into the upper vat *A*. In addition to the drip-tubes, every two vats standing one above the other are connected with a knee. The upper one of these knees *i* issues from the bottom of the vat *A* and enters the vat *B* close to the bottom. The second tube connects in the same manner, *B* with *A*<sup>1</sup>, the third, *A*<sup>1</sup> with *B*<sup>1</sup>, and lowest one, *B*<sup>1</sup> with *C*. Each of these tubes is provided with a cock to allow of the separate vats being connected or disconnected at will. The lowest vat, *C*, is provided with two discharge pipes, one, *i*, at the bottom, and the other, *k*, about 1½ inches higher up.

The five vats rest upon the reservoir, *D*, which serves for the reception of the total quantity of alcoholic liquid which has passed through the apparatus. It is provided with the opening, *g*, which is connected by a rubber tube with the cock, *I*, on *C*. In addition, *D*, is provided with a glass gauge, *p*, which by turning it downward, serves for discharging the fluid from *D*.

The case enclosing the apparatus is constructed of wood and glass, the rubber tube, *g*, passing through the roof of the reservoir *E*, holding the alcoholic fluid. On top the roof is provided with a trap, *m*, which can be opened and closed at will by means of a rope. Below the case is provided with slides *n n*. By opening the slides and the trap, the admission of air can be regulated.

In the commencement of the operation the cock on *E* is opened and the alcoholic fluid is allowed to run through the tube *g*, and the short pipe *h*, until it stands 1½ to 2½ inches deep over the drip-tubes. The cock on the pipe *i* is then closed. The alcoholic fluid now penetrates into the drip-tubes, and runs through them into *B*, which is soon filled so far that the openings of the drip-tubes are reached by the level of the fluid. As the admission of alcoholic fluid continues uninterrupted, one vat after another is filled, and the alcoholic fluid is in the separate drip tubes distributed over a compara-

tively very large surface, being at the same time brought in intimate contact with fresh air.

The admission of alcoholic fluid can readily be regulated, so that on reaching the lowest vat it is converted into vinegar.

When the apparatus is once in operation, the formation of vinegar progresses without interruption, it being only necessary regularly to fill the reservoir with fresh alcoholic fluid, and to draw off the finished vinegar into barrels. If for some reason the operation is to be interrupted, the alcoholic fluid still contained in the vats is drawn off through the respective knees, and the apparatus is filled with water to prevent drying out. When manufacture is to be recommenced, the water is drawn off, and alcoholic fluid admitted.

It is doubtful whether this apparatus possesses advantages over the ordinary generator, since the surface over which the alcoholic fluid is distributed is much smaller than in generators filled with shavings.

*Michaelis' revolving generator* consists essentially of a strong barrel, 3 feet 3 inches in diameter at the widest point, and a space of 3 feet 3 inches between the two bottoms. The barrel rests horizontally upon two supports, so that it can be rolled to and fro upon them. The interior of the barrel is divided by a horizontal lath-grating into two partitions, the upper smaller one being filled with shavings. Below the lath-grating in the bottom of the barrel is a horizontal tube for the admission of air, and above in the side of the barrel a cock for its escape. The alcoholic fluid is poured in close to the lath-grating by means of a funnel, the air-cock is closed and the barrel revolved to allow of the shavings to become thoroughly saturated with the alcoholic liquid. In about 15 minutes the barrel is brought back to its original position, and the air-cock opened. The commencement of the formation of vinegar will soon be recognized by the increase of temperature in the shavings, and the operation is then in full progress.

For the constant continuation of the formation of vinegar, it is only necessary to revolve the barrel for a few moments sev-



eral times a day to saturate the shavings with the alcoholic fluid. The progress of the formation of vinegar is shown by a thermometer placed in the bottom of the upper space, the lowering of the temperature indicating the completion of the process.

The apparatus is cleaned by rinsing the shavings, without taking them from the barrel, with hot water, and filling the barrel with strong vinegar, which is drawn off in 24 hours.

The advantages of this generator consist, according to the inventor, in cheapness of first cost, simple operation, larger yield, saving of alcohol, and better quality of the product.

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## CHAPTER IX.

### AUTOMATIC VINEGAR APPARATUS.

THE principal work to be performed in a vinegar factory consists in pouring at stated intervals the alcoholic fluid into the generators. In a large factory several workmen are constantly engaged in this work, and losses by spilling are unavoidable. Further, it is almost next to impossible always to pour in the same quantity at exactly the same intervals, and sometimes a generator may even be entirely overlooked, and thus remain inactive until the next supply of alcoholic liquid is poured in.

The greatest disadvantage is, however, the interruption for several hours daily, of the formation of vinegar in all the generators, so that, for instance, in a factory working 16 hours a day, one-third of the time is lost. Independently of the small return on the capital invested, these interruptions are accompanied by many other conditions injurious to the regular running of the factory.

The greatest of these evils is that with the cessation of the supply of alcoholic fluid the propagation of the vinegar fer-

ment diminishes and finally ceases altogether. Further, the development of heat in the interior of the apparatus at the same time ceases and the temperature is reduced several degrees, this phenomenon appearing even in factories provided with the best heating apparatus and keeping up a constant temperature in the workroom during the night.

In the morning when work is resumed, it is in most cases necessary to vigorously air the apparatus by opening all the draught holes, in order to gradually restore the temperature to the required degree, and it will be some time before the apparatus again works in a normal manner.

The vinegar ferment, however, is very sensitive to changes of temperature, as well as to the concentration of the nourishing substances surrounding it, and there can be no doubt that its propagation is prejudiced by the continuous variations of temperature to which it is exposed during the interruptions of several hours a day. That such is actually the case is shown by the fact that the quantity of vinegar ferment formed in the generators is small as compared with that which, under conditions favorable to the ferment, forms in a short time upon alcoholic liquids.

Besides the debilitation of the vinegar ferment and the consequent disturbance in the regular working of the factory, the repeated reduction of the temperature in the generators has the further disadvantage that, besides the vinegar ferment, other ferments for whose development a low temperature is more favorable may be formed, and these ferments may increase to such an extent as to entirely suppress the vinegar ferment. There can scarcely be a doubt that the many apparently inexplicable disturbances in the working of the generators, such as their remaining cool notwithstanding an increased current of air, the vinegar becoming suddenly weaker, or the entire cessation of its formation, find their easy explanation in the daily interruptions lasting for hours in the regular working of the factory.

Besides the increase in the capacity of the factory, disturb-

ances are, therefore, less likely to occur where the work is carried on uninterruptedly, but in order to do this there must also be a corresponding increase in the number of workmen employed in pouring alcoholic liquid into the generators.

By the use of simple automatic contrivances for the regular pouring out of the alcoholic liquid, the number of workmen employed in a vinegar factory can, however, be reduced to the attendance required for looking after the heating apparatus, raising the alcoholic liquid to a certain height, and an occasional control of the temperature in the interior of the generators. A factory thus arranged requires but little attendance, as when once in good working order it may be left to itself for many hours without the occurrence of any disturbance.

According to the characteristics which distinguish the different constructions of continuously working apparatus from each other, they may be divided into two principal systems, viz., into those with an uninterrupted, and those with a periodical, pouring out of the alcoholic liquid, but in either case the latter has to be brought into a reservoir placed at a certain height above the generators.

#### CONTINUOUSLY ACTING APPARATUS.

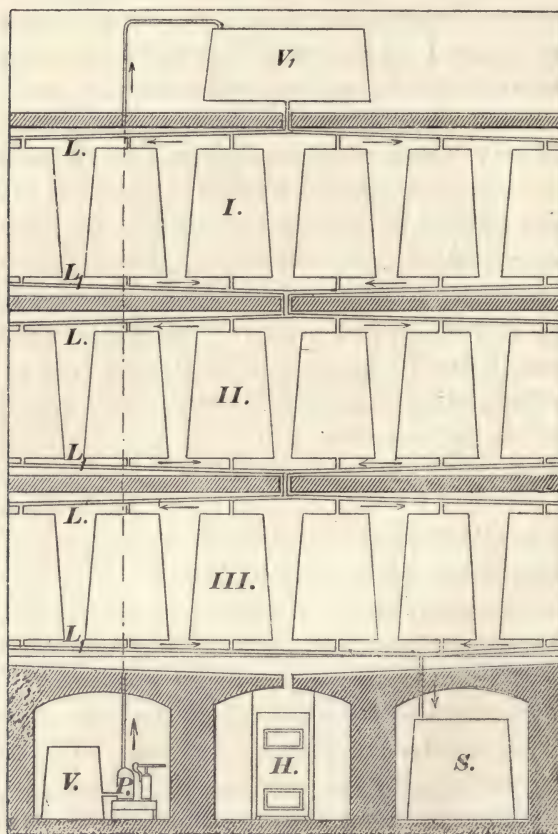
*The Terrace System.*—The alcoholic liquid, as is well known, cannot be converted into finished vinegar by passing once through the generator, a repeated pouring into several, generally three, different generators being required. To avoid the necessity of raising the alcoholic liquid three times, three rows of generators have been arranged one above another, so that the alcoholic liquid coming from a reservoir placed at a higher level flows first into the uppermost generator, and passing through this, runs directly into the second, and from there into the third, which it leaves as finished vinegar. Fig. 27 shows a vinegar factory arranged according to this system, *I, II, III*, representing the three rows of generators placed one above another, *V*<sup>1</sup> the reservoir for the alcoholic liquid, *P* the arrangement for pumping the alcoholic liquid into the reser-



voir, *V* the distributing vessel for the alcoholic liquid, *S* the collecting vessel for the finished vinegar, *H* the heating apparatus for the entire establishment.

For a uniform supply of alcoholic liquid to the generators

FIG. 27.



standing on the same level a conduit, *L*, from which the alcoholic liquid flows into each generator, runs above the uppermost row. Another conduit, *L*<sup>1</sup>, common to all the generators, serves for the reception of fluid (finished vinegar) running off from the lowest row, and conducts it to the collecting ves-

sel, S. The arrows indicate the course the alcoholic liquid has to traverse.

From all appearances the arrangement of a factory according to the above-described system would be most advisable, there being actually nothing to do but to raise the alcoholic liquid once and to remove the finished vinegar from the collecting vessel. In practice, however, this so-called terrace system presents many difficulties not easily overcome, the greatest undoubtedly being the solution of the heating problem. Experience shows that the temperature in a generator must be the higher the more acetic acid the alcoholic liquid contains. According to this, the highest temperature should prevail in the lowest series of generators (*III*, Fig. 27) and the lowest in the uppermost (*I*).

But in practice just the reverse is the case even with the use of the best heating apparatus, the highest temperature prevailing in *I* and the lowest in *III*, as, according to natural law, the warm air being specifically lighter than the cold constantly strives to ascend.

To overcome this drawback nothing can be done but to place the series *I*, *II* and *III* of the generators in as many different stories entirely separated from each other, or, in case there is a central heating apparatus in the cellar, to correctly distribute the warm air in the separate stories by suitably arranged registers. The solution of this problem offers no insuperable difficulties, but requires the arrangement of the entire factory to be carefully planned in accordance with the laws of physics.

An unavoidable drawback of the terrace system is the costliness of the factory building, and, finally, that a disturbance occurring in one of the generators must simultaneously affect two others of the vertical series, which must necessarily remain idle until the disturbance is removed. Considering all the disadvantages connected with the terrace system, though it is seemingly so suitable, it is but little adapted to practice, it being much preferable to place all the generators on the same level and to divide them into three groups, each of which is

provided with a reservoir for the alcoholic liquid and a collecting vessel.

The mode of working according to this system is as follows: The alcoholic liquid is pumped into a reservoir, from which it passes through group *I* of generators and collects in a vessel. From the latter it is pumped into a second reservoir placed on the same level with the first, and runs through group *II* of generators into another collecting vessel; from there it is again pumped into a third reservoir, and after passing through group *III* of generators finally collects as finished vinegar in a third collecting vessel.

Though the arrangement of all the generators on the same level renders it necessary to raise the alcoholic liquid three times, it would seem more suitable than the terrace system for the following reasons: 1. By a suitable regulation of the heating apparatus the required temperature can be readily maintained in the separate groups of generators. 2. In case of a disturbance in one of the groups, the generator in question can be left out without causing an interruption in the work of the other groups. 3. The power required to pump the alcoholic liquid three times into the reservoirs  $V_1$ ,  $V_2$ , and  $V_3$ , is not much greater than that which has to be used to raise it to the height of the reservoir in factories arranged according to the terrace system. 4. Notwithstanding the greater area required, the erection of a one-story factory is less expensive than that of a three-story building with complicated heating apparatus and very strong, solid floors, which are required on account of the great weight of the generators.

The uniform distribution of the alcoholic liquid into each generator is very simple in factories arranged according to the terrace system, and can be effected in the following manner:

The false heads are fitted water-tight in the generators; they are provided either with narrow holes alone, or with apertures loosely filled with cotton-wick, pack-thread, etc. The pipes ascending from the vinegar-forming space, which is filled with shavings, are inserted water-tight in the false bottoms.



On the reservoir containing the alcoholic liquid is a spigot which can be accurately adjusted, and is securely connected with the conduit leading to the separate generators. At the place on the conduit where the alcoholic liquid is to be introduced into the generator is a discharge-pipe also provided with a spigot.

When the factory is to be put in operation the reservoir is first filled with alcoholic liquid, the spigots on the several generators being entirely open, but the principal spigot closed. Now, by suddenly opening the latter, the air in the conduit is expelled by the alcoholic liquid flowing in, and the latter rushes in a full stream from the spigots connecting the conduit with the generators. These spigots are then closed so far that only the quantity of alcoholic liquid required for the regular process of the formation of vinegar can enter the generators. To prevent the force of pressure from varying too much in the conduit by the lowering of the level of the fluid in the reservoir, it is recommended to give the latter only a slight height but a large bottom surface.

From the lower portion of the uppermost series of generators the alcoholic fluid then gradually reaches through a pipe the false bottoms of the next series, and from this the lowest series, from which it runs off as finished vinegar into the collecting vessel.

It will readily be seen that some time for experimenting is required before a factory arranged according to this system can be brought into regular working order, it being necessary to test the fluids running off from the different groups of generators as to their contents of acetic acid in order to find out whether too much or too little or just enough alcoholic liquid reaches the generator, so that the liquid running off from the lowest series contains no alcohol and may be considered as finished vinegar. Any fault in the working of the generators can in this case be overcome by a corresponding adjustment of the spigots so as to regulate the influx of alcoholic liquid.

Theoretically no more simple or convenient process for

making vinegar than the terrace system could be devised. Provided the spigots supplying the separate generators be once correctly adjusted and the temperature of the different stories suitably regulated, it is only necessary constantly to supply the reservoir with alcoholic liquid, and the heating apparatus with fuel, in order to carry on the work for any length of time desired. The disadvantages connected with this system having been already explained need not be further referred to.

*Lenze's chamber generator* : \* This apparatus is a Schuetzenbach generator of logically improved construction, and not, as might be supposed at the first glance, an arbitrary modification of the exterior shape. Its construction is based upon the following principles :

1. Saving of space and volume.
2. Simplification of the work and facility of control.
3. Utilization to the best advantage of the square fermentation-surface.
4. The process is carried on without being separated by partitions in many isolated narrow columns of shavings.
5. Surface-fermentation with little height.
6. Large producing capacity.

The average height of the apparatus is about 7 feet. It is rectangular in form, is built entirely of wood, no hoops whatever being used, and is of solid and massive construction. It is made in three sizes. Fig. 28 shows a No. 3 apparatus with a base of about 107 square feet, length 10 feet, width 6 feet, and a capacity of producing 20 to 25 quarts of 13 per cent. vinegar per  $10\frac{1}{2}$  square feet of shavings-surface (base of generator).

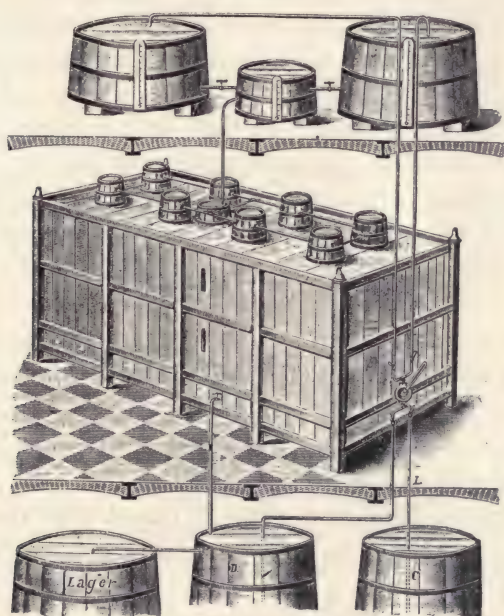
The alcoholic liquid is periodically supplied at fixed intervals, but the operation may also be carried on continuously day and night.

The attendance of the apparatus is entirely mechanical, by means of a pump operated by hand or power. Losses of material by spilling or otherwise are impossible since the alcoholic liquid moves in closed tin pipes and the finished vinegar is conveyed in the same manner.

\* J. Lenze, Iserlohn, Westfalen, Germany.

The apparatus is furnished with a lath-bottom and a perforated head, the intermediate space being packed with beech shavings. The air-holes are between the actual bottom and the lath-bottom and the air-outlets below the perforated head. The alcoholic liquid is very uniformly distributed over the entire large surface of shavings by the perforated head. The latter is tightly covered with cloth, divided into square fields,

FIG. 28.



and so secured that it cannot warp or get out of position. Notwithstanding its large superficial area the perforated head is the coolest place in the apparatus and this evidently contributes towards reducing the loss by evaporation to a minimum, and such loss can be still further limited by the use of a cover fitting almost air-tight. The upper layer of shavings is also not impaired by higher degrees of heat, because the ascending air which has been heated and exhausted, is con-



stantly cooled by the pourings of alcoholic liquid and partly condenses on the lower surface of the perforated head, and thus cooled, escapes through a pipe-system below the perforated head.

The mode of operating such an apparatus and its attendance is illustrated by Fig. 28.

The alcoholic liquid is contained in the vat *C* and a sufficient quantity of it is by means of the pump *P* conveyed to the vat *A*, where it is diluted to a weak wash. When the operation is carried on with back pourings, the vat *B* contains the vinegar; otherwise it is omitted. The intermediate vat *E* effects automatically by suitable contrivances the measuring off of the separate pourings, so that after pumping the entire quantity of alcoholic liquid required for one day, the actual labor is finished, which requires about one hour's consumption of time and power, no matter whether one or several apparatuses are operated, larger pumps being used in the latter case.

For the accurate control of the operation a contrivance is provided which indicates in the office whether and at what time the separate pourings have been effected.

The product running off from the apparatus collects in the course of the day in the vat *D*. When the latter is full, the overflow passes through a pipe to the storage-vat. By this arrangement the danger of any of the vats running over is excluded.

*Plate Generator.*—This generator, patented by Dr. Bersch, of Vienna, Austria, is so arranged as to render the formation of aldehyde as well as the destruction of acetic acid already formed impossible, and the loss by evaporation is reduced to a minimum. As will be seen from the description, the arrangement of the apparatus is such that on all portions of the surface of the plates air and alcoholic liquid are in undisturbed contact. Hence the formation of vinegar takes place constantly and the regulation of the current of air can be effected with the utmost accuracy. Since the effective surface

of each apparatus, *i. e.*, the surface upon which the formation of vinegar actually takes place, is more than 10,764 square feet, the performance of this generator is extremely large, surpassing by far that of a generator packed with shavings.

This generator is provided with a contrivance which automatically attends to the pouring-in of alcoholic fluid with the regularity of clock-work, and thus the work of a factory using a large number of generators can be done by a single workman, he having nothing else to do than to fill once a day the reservoir for alcoholic fluid.

In its most recent construction the plate-generator consists of a vat filled inside with layers of extremely thin plates of wood arranged in such a manner that the separate layers are fixed crosswise at right angles one above the other. Since every two plates in the layers lying alongside each other are kept apart by prismatic wooden rods, fluid can run down on both sides of the plates, and air ascend undisturbed between them.

Since the total surface of the wooden plates in a generator about  $8\frac{1}{4}$  feet high is more than 10764 square feet, and formation of vinegar takes place uninterruptedly upon this entire surface, the efficacy of the plate generator as regards producing capacity is the highest attainable.

Through a pipe, open at both ends, in the bottom of the generator, air is admitted to the interior. The upper portion of this pipe is furnished with a hood to prevent fluid from dropping into it, and the lower opening is covered with fine gauze to exclude the entrance of vinegar lice (vinegar mites).

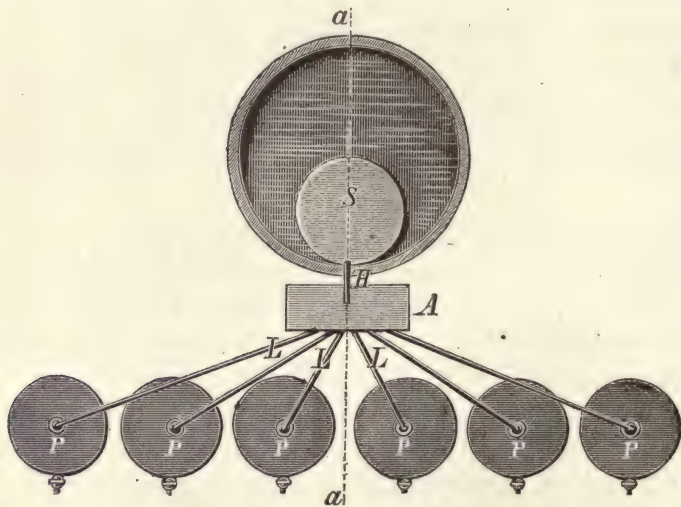
The strength of the current of air in the generator is regulated by a register-bar in the cover of the apparatus, in which is also fixed a thermometer. In the commencement of the operation the register-bar is so set that the thermometer indicates the temperature—about  $91^{\circ}$  to  $93^{\circ}$  F.—suitable for the formation of vinegar. So long as alcoholic liquid runs in and the temperature of the workroom remains the same, the same temperature will be indicated by the thermometer, be-

cause in equal periods of time the same quantity of vinegar will always be formed and a quantity of heat corresponding to it developed.

The uniform distribution of the alcoholic fluid in the form of very fine drops over the plates in the interior of the generator is effected by a sparger fixed over the uppermost layer of plates.

The apparatus, like every other generator, can be charged by pouring in the alcoholic liquid by hand. However, to make it entirely independent of the workman, and especially

FIG. 29.



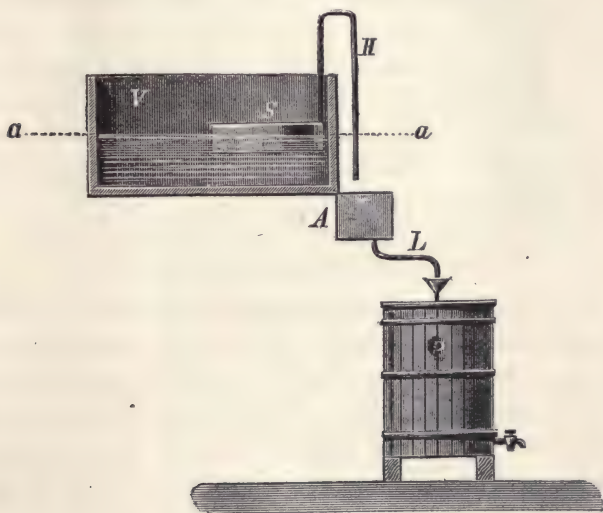
to keep it working regularly day and night without interruption, it is provided with an automatic pouring contrivance. This contrivance consists of a vat of such a size as to be capable of holding the fluid required for supplying for 24 hours one generator or a group of two, three, four or more generators. In this supply-vat floats in a suitable guide a wooden float-gauge, which rises and sinks with the level of the fluid. To this float-gauge is secured a siphon, the longer leg of which is furnished with a checking contrivance which has to be



accurately regulated. By shifting this checking contrivance, the quantity of fluid discharged in a certain unit of time, for instance, in one hour, can be determined. Since the siphon sinks with the level of the fluid, and its length remains unchanged, the fluid always runs off under the same pressure.

The liquid running from the siphon passes into a distributing vessel underneath. The latter should be of sufficient capacity to hold the total quantity of liquid required for one

FIG. 30.



pouring upon all the generators in a battery. If, for instance, every generator in a battery of twenty-four is to receive a pouring of 3 quarts, the distributing vessel should have a capacity of at least  $3 \times 24 = 72$  quarts. The automatic pouring contrivance is fixed in the distributing vessel. When the latter contains the quantity of alcoholic liquid required for one pouring for a determined number of generators, the time fixed between every two pourings has elapsed. The automatic pouring contrivance then opens the distributing vessel and the alcoholic liquid passes through the conduits to the

generators. When the distributing vessel has been emptied, the discharge-contrivance closes automatically, the distributing vessel is filled within the determined time, and is again emptied when this time has elapsed. The automatic distributing contrivance thus continues working without interruption so long as liquid is contained in the supply-vat. If the latter is of sufficient capacity to hold enough alcoholic liquid for 24 hours, it is only necessary to fill it in the morning.

Figs. 29 and 30 show the arrangement of the separate parts of a plant for automatically working plate generators. Fig. 29 is a view from above and Fig. 30 a side view. *V* is the supply-vat for the alcoholic liquid, *S* the float-gauge to which is secured the siphon *H*. *A* is the automatic distributor, and *I* the conduits conveying the alcoholic liquid to the separate plate-generators *P*. The dotted line *aa* represents the level of the fluid in the supply-vat.

#### PERIODICALLY WORKING APPARATUS.

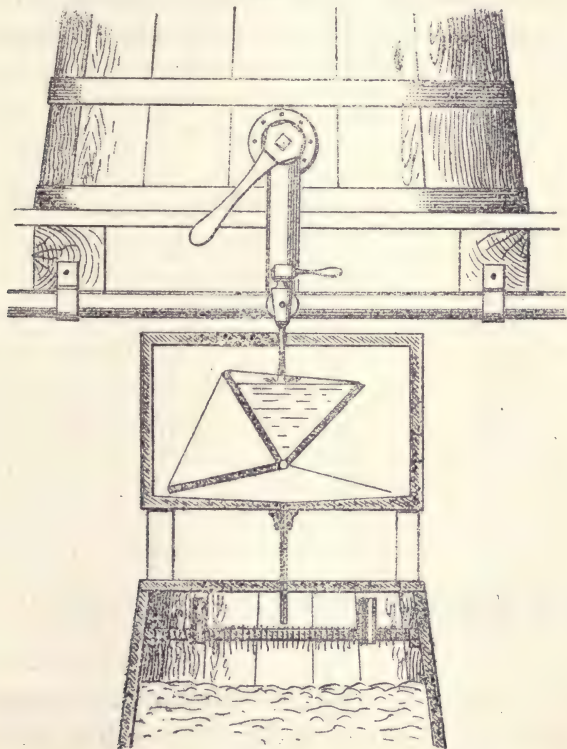
*The Three-group System.*—In the second system of automatic generators it has been sought to imitate the ordinary working of a vinegar factory by providing the apparatus with certain mechanical appliances which allow of the distribution at certain stated intervals of any desired quantity of alcoholic liquid into the generators. The term “periodical” may be applied to this system of automatic apparatus.

The mechanical appliances used for the purpose of admitting at certain intervals a fixed quantity of alcoholic fluid into the generator may be constructed in various ways, the tilting trough, shown in Figs. 12 and 13, p. 49, being an example. By a modification of the apparatus, as shown in Fig. 31, any desired quantity of fluid can with its assistance be at certain intervals admitted to the generator. The fluid may be either poured out upon the false head, or, what is more suitable for its better distribution, used for feeding a sparger.

As seen from the illustration a prismatic box with a bottom formed of two slightly inclined planes, stands at a suitable

height over each generator. In this box a tilting trough is placed so that its axis of revolution runs parallel with the line formed by the two bottom surfaces of the box. On the point of contact of the two, a pipe is inserted which extends to the false head or to the funnel of the sparger. Above the box is a spigot connected by a pipe with a reservoir for the alcoholic

FIG. 31.

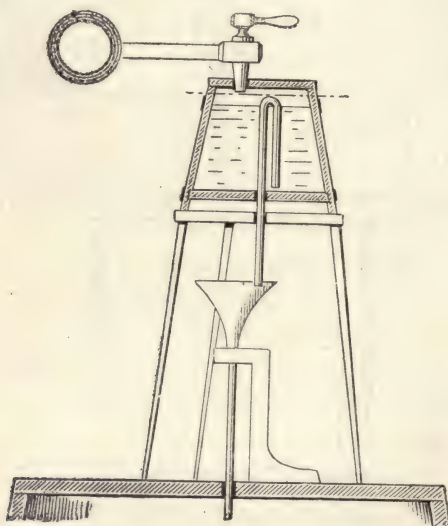


fluid placed at a higher level. This reservoir serves for supplying a large number of generators, and can be shut off by a carefully adjusted spigot. From the latter a vertical pipe leads to the conduit running in a horizontal direction over the generators. The pipe is provided with small spigots which discharge the fluid into the tilting troughs.



By giving each tilting trough such a capacity that, for instance, each partition holds 5 quarts, and adjusting the spigot so that 30 minutes are required for filling one partition, the trough will, at the expiration of this time, tilt over and empty the fluid upon the inclined planes. From here it runs into the sparger, and setting the latter in motion is poured in the form of a fine spray over the shavings. Since the other partition of the tilting trough has the same capacity, as the first, and the quantity of the alcoholic fluid remains the same, the

FIG. 32.



trough will, after the expiration of 30 minutes, again tilt over, and again empty 5 quarts of fluid, this being continued as long as the reservoir contains any fluid.

In place of the tilting trough the so-called "siphon-barrel," Fig. 32, may be used for effecting the discharge of a certain quantity of fluid at a stated interval. In a spherical vessel placed at a higher level than the edge of the funnel of the sparger is a siphon, the longer leg of which passes through the bottom of the vessel into the funnel. On the edge of the vessel

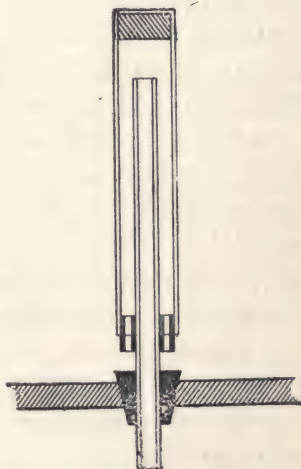
is a spigot which is connected with the pipe conveying the fluid, and so adjusted that within a previously determined space of time the vessel is filled with fluid up to the height indicated by the dotted line. As soon as the fluid reaches that height, action of the siphon commences, and the content of the vessel runs through the longer leg into the funnel of the sparger until its level is sunk to the edge of the shorter leg. The action of the siphon then ceases until the vessel is again filled up to the line, when it recommences, and so on.

The siphon of bent glass tubes being very liable to breakage, it is frequently replaced by the so-called bell-siphon, the arrangement of which is shown in Fig. 33. It consists of a glass tube which forms the longer leg of the siphon, while a glass cylinder secured to this tube by means of a perforated cork, represents the other leg. The action of this siphon is the same as the other.

In working with automatic apparatus, fixed quantities of fluid being at stated intervals introduced, provision for the reception of the fluid must be made in the apparatus itself, or for its being conducted to a special reservoir at the rate at which it trickles from the shavings. In the first case the space beneath the false bottom must be of sufficient size to receive the fluid passed through the apparatus in a certain time. This time being suitably fixed for 12 hours, the apparatus can during this time work without further attendance, so that the required space beneath the false bottom can be calculated by multiplying the number of pourings with the quantity of fluid poured in at one time.

Example:—The generator receives at intervals of 30 minutes a pouring of 5 quarts, hence in 12 hours 24 pourings of

FIG. 33.



5 quarts each = 120 quarts. The space beneath the perforated false bottom must therefore be of sufficient capacity to receive up to the height of the draught-holes at least 120 quarts of fluid.

As will be seen from the following general description of a vinegar factory, arranged according to the automatic principle, it is decidedly preferable to arrange the generators so that the fluid trickling from the shavings is at once conducted to a collecting vessel.

*Arrangement of a Vinegar Factory Working According to the Automatic Principle.*—As previously stated, it is not possible to convert all the alcohol contained in the liquid into acetic acid by one pouring; only a portion of the alcohol being converted, and this semi-product is brought into a second generator, and, if the liquid used is very rich in alcohol, into a third. In the second apparatus another portion of the alcohol is converted into acetic acid, and the process finished in the third.

It being in all cases advisable to prepare vinegar with a high percentage of acetic acid, most manufacturers now pass the alcoholic liquid successively through three generators. In practice it is recommended to place the generators which are to receive alcoholic liquid of the same content of acetic acid alongside each other, which leads naturally to the division of the generators into three groups. If, for instance, a factory contains 48 generators, each group contains 16; group I is charged with freshly prepared alcoholic liquid; the generators of group II contain the alcoholic liquid which has already passed through those of group I, and group III is charged with the fluid yielded by group II.

Besides the easy control of the work, this arrangement into groups has another advantage. The generators in which the last remnants of the alcohol of a quite strong fluid are to be converted into acetic acid are best kept at a somewhat higher temperature; and with a suitably arranged heating apparatus and the eventual use of curtains by which the workroom can be divided at will into two or three partitions, it can be readily



arranged to convey somewhat more heat to the second group of generators and the greatest quantity of it to the third.

The height of the actual workroom of the factory should not be greater than required by that of the generators. The reservoir is placed under the roof of the workroom, while the collecting vessels are either sunk in the floor or placed in the cellar.

Below is given a description of a periodically working establishment with 24 generators. The generators are arranged in three groups, I, II, and III, the following articles belonging to each group :—

8 generators ;

1 reservoir ;

1 collecting vessel ;

8 apparatuses for the distribution of the alcoholic liquid into the generators ;

Conduits for the alcoholic liquid to be poured in ;

Conduits for the alcoholic liquid running off.

For the three groups in common :—

A pump to convey the alcoholic liquid from the collecting vessels to the reservoirs.

A flue for the conveyance of warm air from the heating apparatus in the cellar and for its distribution in the workroom.

An apparatus for heating the alcoholic liquid.

The three reservoirs rest upon the joists of the ceiling of the workroom, each being enclosed by a small chamber constructed of boards which are papered. In the floor of each chamber is a man-hole for access to the reservoir. The man-holes should not be furnished with doors, it being of importance that the reservoirs should constantly be surrounded by warm air which ascends through the man-holes. To prevent loss by evaporation the reservoirs should be provided with well-fitting covers.

To retain solid bodies such as shavings, flakes of mother of vinegar, etc., which might eventually obstruct the fine aper-

tures in the false head or sparger, a filter is placed on the end of the pipe through which the alcoholic liquid passes into the reservoirs. A suitable filter for the purpose is a horse-hair sieve containing a linen bag, the latter being from time to time replaced by a new one.

The conduits for the conveyance of the alcoholic liquid to the distributing vessels and from there to the generators are best constructed of thick glass tubes, the connection of two pieces being effected by pieces of rubber hose pushed over the ends and secured with twine.

Each generator may be furnished with a vessel containing the automatic arrangement, it being, however, in this case necessary to provide for each a special conduit from the reservoir, which for a factory containing a large number of generators is rather expensive. Hence it is recommended to use for each group only one or at the utmost two distributing vessels, and from them to extend smaller conduits to the separate generators. Each of the principal conduits is provided, at the place where it enters the distributing vessel, with a spigot, which is adjusted for the discharge of a certain quantity of alcoholic liquid. If, as above mentioned, every generator is to receive a pouring of 5 quarts of alcoholic liquid every 30 minutes, the distributing vessel serving for a group of 8 generators must have a capacity of 40 quarts, and the spigot has to be so adjusted that exactly this quantity passes through it in 30 minutes.

The discharge-pipe of the automatic arrangement must enter a space in which are inserted eight pipes having the same diameter, which conduct the alcoholic liquid to the separate generators. By this arrangement all the generators receive simultaneously a pouring of an equal quantity of fluid which either sets the sparger in motion or gradually trickles through the apertures in the false head. The alcoholic liquid which has passed through the generators collects either in the space under the false bottom or runs directly through conduits to the collecting vessels.

The conduits placed before the discharge apertures of the generators are intended to conduct the alcoholic liquid to the reservoirs, and there being no pressure of fluid in them they might be merely open gutters. For the sake of cleanliness and to avoid losses by evaporation it is, however, advisable to use glass tubes for the purpose. At the places where the discharge-pipes of the generators are located, the connection of two glass tubes is effected by a wooden joint with an aperture on top in which is placed a glass funnel. For collecting vessels for the alcoholic fluid running off from the generators of one group, vats provided with lids are used. They have to be placed so low that some fall can be given to the conduits, and in each of them is a pipe provided with a spigot, which serves as a suction-pipe for the pump intended to raise the alcoholic fluid.

The manner of working in a factory thus arranged is as follows: \* The collecting vessel C<sub>I</sub> serves for the preparation of the alcoholic liquid, which is then pumped into the reservoir R<sub>I</sub>, from whence it runs through the first group of generators, G<sub>I</sub>, to the collecting vessel, C<sub>II</sub>. From this it is pumped into R<sub>II</sub>, and runs through the second group of generators, G<sub>II</sub>, into the collecting vessel C<sub>III</sub>. On being pumped up the third time it runs from the reservoir R<sub>III</sub> through the third group of generators G<sub>III</sub>, and passes as finished vinegar either into a fourth collecting vessel or is at once conducted into storage barrels.

The distance the alcoholic liquid has to be raised from the bottom of the collecting vessels to the reservoir amounting to not more than from 23 to 25 feet, an ordinary suction-pump may be used for the purpose, though a forcing pump is better, it doing the work more rapidly. The pump must be constructed of material entirely indifferent to acetic acid (wood, glass, hard rubber, tin, or thickly silvered metal).

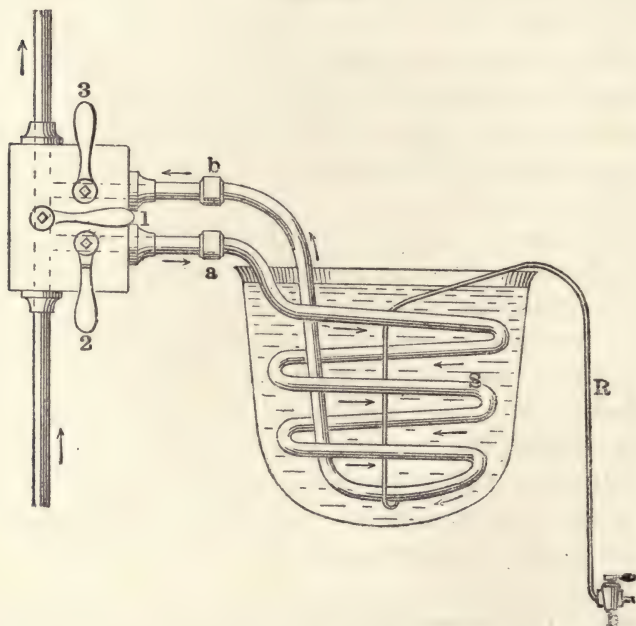
\* To avoid repetition the collecting vessels are designated: C<sub>I</sub>, C<sub>II</sub>, C<sub>III</sub>; the reservoirs R<sub>I</sub>, R<sub>II</sub>, and R<sub>III</sub>; the groups of generators G<sub>I</sub>, G<sub>II</sub>, G<sub>III</sub>.



Any metallic vessels used in the factory should be of *pure* tin, *i. e.*, unalloyed with other metals, it being the only metal entirely indifferent towards acetic acid, but unfortunately it is too soft to be suitable for the construction of pumps.

The pump is generally located in the immediate neighborhood of the collecting vessels, and the three branches of its suction pipe pass into the latter. When one of the collecting

FIG. 34.



vessels is to be emptied, the spigot of the branch pipe entering it is opened and the spigots of the other branch pipes are closed.

Ordinary well or river water being used in the preparation of the alcoholic liquid, the temperature of the latter does not generally exceed  $54^{\circ}$  F., and if it were thus introduced into the generators acetification would be very sluggish until the temperature rises to above  $68^{\circ}$  F. Independently of the loss

of time, there would be the further danger of injuring the development of the vinegar ferment ; hence it is necessary to heat the alcoholic liquid to the temperature required. This is best effected by passing it through a coil surrounded by hot water. Fig. 34 shows an apparatus especially adapted for heating the alcoholic liquid. In a copper or iron boiler filled with water, which can be heated from below, is a coil, *S*, of pure tin ; it enters the boiler above at *a* and leaves it at *b*, so that the place of inflow is at the same level with that of outflow. With this form of construction the coil of course remains always filled with liquid, which with the use of pure tin is, however, of no consequence ; besides, this can be remedied by placing on the lower coil a narrow pipe, *R*, which projects above the edge of the boiler and is bent like a siphon. By opening the spigot *h* the fluid contained in the coil runs off through *R*.

The rising pipe of the forcing-pump is provided with an arrangement by which the alcoholic liquid can be brought either directly from the collecting vessel into the reservoirs, or first forced through the heating apparatus. It consists of a prismatic wooden body provided with three spigots. By closing spigots 2 and 3 and opening 1, the alcoholic liquid is immediately conveyed from the collecting vessels to the reservoirs. By closing spigot 1 and opening 2 and 3, which are connected by short pieces of rubber hose with the ends of the coil, *S*, the alcoholic liquid forced upward from the collecting vessels by the pump must pass through the heating coil, and after being heated it returns to the rising pipe, which conveys it to the reservoirs. The arrows in the illustration indicate the course of the alcoholic liquid has to traverse when spigots 2 and 3 are open and 1 closed.

The diameter and length of the tin coil depends on the quantity of fluid which is to pass through it, though one with a clear diameter of 12 to 14 inches and a length of 23 to 26 feet will, as a rule, suffice. Besides by slower or quicker pumping the fluid can be forced with less or greater velocity through the coil and correspondingly more or less heated.

The walls of the coil should be as thin as possible so as to yield heat rapidly.

The heating of the alcoholic liquid, of course, can also be effected by heating one portion more strongly than necessary and reducing it to the required temperature by mixing with cold fluid. In working, however, with a fluid containing living vinegar ferment—and such, as will be explained later on, is claimed to be already contained in freshly prepared alcoholic fluid—care must be had not to heat the liquid above  $120^{\circ}$  F., this temperature being destructive to the ferment.

## CHAPTER X.

### OPERATIONS IN A VINEGAR FACTORY.

*Acetification of the Generators.*—The object of acetification is to thoroughly saturate the filling material—shavings, charcoal, etc.—of the generators with vinegar and to cause the development of the vinegar ferment upon it. The generators are first filled with the filling material, the false heads or the spargers are next placed in position, and the temperature of the workroom is brought up close to  $86^{\circ}$  F. For acetification, *i. e.*, saturating the shavings, vinegar of the same strength, *i. e.*, with the same content of acetic acid as that which is to be prepared in the generators, is used. For every  $35\frac{1}{2}$  cubic feet of the space filled with shavings or charcoal are required for complete acetification the following quantities of vinegar:

For shavings loosely poured in.....	$60\frac{3}{4}$ to $71\frac{1}{2}$ gallons.
For shavings piled up one alongside the other ..	90 to $105\frac{1}{2}$ gallons.
For charcoal, the size of a walnut .....	$142\frac{1}{2}$ to $211\frac{1}{2}$ gallons.

The value of the vinegar used for acetification has to be considered as dead capital.



The first vinegar running off from the generators is not only considerably weaker than that used for acetification, but, notwithstanding the previous lixiviation of the wood, has a disagreeable taste so as to render it unfit for the preparation of table vinegar, and can only be utilized, for instance, in the preparation of acetate of lead, etc. When the vinegar running off exhibits a pure taste, it is collected by itself and later on converted into a product of greater strength by mixing it with alcohol and passing again through the generators. By this saturation of the shavings with vinegar, the vinegar ferment locates in abundance upon the surface of the shavings, and the generators are fit for the formation of vinegar.

Regular production, however, can be commenced only gradually, which may be illustrated by an example as follows:

At first, for instance, alcoholic liquid is introduced only once a day, either early in the morning or in the evening. In about eight days, or under certain conditions even later, the temperature in the generators has risen to from  $86^{\circ}$  to  $95^{\circ}$  F., and alcoholic liquid may now be introduced twice daily, for instance, early in the morning and in the afternoon. The fact that the generator is working is recognized by the increased temperature and by the flame of a candle held near a draught-hole being drawn inwards. After eight to fourteen days more the thermometer shows  $96^{\circ}$  to  $98^{\circ}$  F., and then alcoholic liquid is introduced three times daily, for instance, early in the morning, in the forenoon and in the afternoon, whereby the temperature rises to  $102^{\circ}$  or  $104^{\circ}$  F. If now the vinegar running off shows the desired strength, the generators are in good working order, and are subjected to the regular treatment.

*Accelerated Acetification.*—By closely considering the process which must take place in acetification and the first stage of the operation, it will be plainly seen that the above-described method cannot be called a rational one, there being a waste of time as well as of material, and the commencement of regular working being largely dependent upon accident.

The object of acetification is, as previously stated, first to thoroughly saturate the shavings with vinegar and next to develop the vinegar ferment upon them. This can, however, be attained in a more suitable and a quicker manner than by the above process.

Air-dry wood contains on an average 20 per cent. of water, and during acetification this water must be gradually replaced by vinegar; hence the vinegar trickling from the generators will remain poor in acetic acid and rich in water until the shavings are entirely saturated with pure vinegar and the water has been expelled.

The removal of the water from the shavings and its substitution by vinegar are effected by osmose, *i. e.*, the cells of the wood surrounded by vinegar yield a fluid consisting of water and extractive substances of the wood and absorb sufficient of the exterior fluid until both liquids have the same composition. Now, by pouring only a small quantity of vinegar at one time over the shavings in the generator, as is done in acetification according to the old method, the course of the process is very slow, 14 days or more, as already mentioned, being required before the vinegar running off shows no longer a change in its concentration.

In a generator in a stage of acetification an uninterrupted, though slight, current of air upwards takes place, since even with the use of the best heating apparatus the air in the upper layers is warmer than in the lower. This current of air becomes stronger with the development of larger quantities of vinegar ferment and causes a large absolute loss of vinegar. The greater portion of this loss must be set down as being due to evaporation, which must be considerable on account of the great surface over which the vinegar is distributed, and the smaller portion, to consumption by the vinegar ferment.

By placing the shavings in vinegar the above-described process of substitution of vinegar for the fluid contained in the cells of the wood takes place very quickly, and, theoretically, it would therefore seem to be advisable to follow the same

course on a large scale, *i. e.*, to saturate the shavings with vinegar before placing them in the generators. By using artificially dried shavings (see p. 54) the saturation is effected in the course of a few hours, the dry woody tissue absorbing the fluid like a sponge.

The shavings, while still hot, are brought into a vat and covered with the vinegar to be used for acetification. In about 12 hours they are thoroughly saturated. The excess of vinegar is drawn off through the tap-hole in the bottom of the vat, and having absorbed neither water nor extractive substances from the steamed and thoroughly dried shavings can be immediately re-used for the saturation of another portion of shavings. The saturated shavings are at once used for filling a generator, and the latter, which may now be considered as completely acetified, can at once be employed for the process of the formation of vinegar according to the method described below.

Instead of in a vat, the shavings can also be saturated directly in the generator. For this purpose the shavings, after having been artificially dried, are immediately brought into the generator, and vinegar is poured over them either by means of the false head or the sparger until a considerable quantity has accumulated in the space below the false bottom. This accumulation is then drawn off and again poured over the shavings, this being continued until they are thoroughly saturated, which is effected in a comparatively short time.

*Induction of the Operation with an Artificial Culture of Vinegar Ferment.*—In the process of accelerated acetification of the generators no development of vinegar ferment can of course take place, since by heating the shavings to about 212° F. any fermenting organisms accidentally adhering to them are destroyed. The vinegar ferment increases with astonishing rapidity provided it finds nutriment suitable for its development. Vinegar is, however, a very poor material for this purpose, and this is very likely the reason why weeks are required before production can be commenced in generators



acetified according to the old method. The ferment can, however, be so rapidly propagated in the generator that production can be commenced almost immediately after acetification is complete.

For this purpose a method similar to that employed in the manufacture of alcohol and yeast has to be pursued and vigorous ferment obtained by cultivation. As previously mentioned, the ferment causing acetic fermentation is widely distributed throughout nature and is most abundantly found in the air of thickly populated regions.

The "pure culture" of the vinegar ferment, *i. e.*, in which no other than the desired ferment is developed, is not difficult, it being only necessary to prepare a fluid especially adapted for its nutriment and allow it to stand at a suitable temperature in order to obtain in a few days a vigorous growth produced by a few individual germs reaching the fluid from the air. The best fluid for the purpose is one which contains, besides a large quantity of water—about 85 to 90 per cent.—a certain amount of alcohol and acetic acid, and very small quantities of nitrogenous substances and mineral salts. Hence its preparation is not difficult, it being only necessary to mix ordinary vinegar and alcohol in suitable proportions and add a small quantity of a fluid containing nitrogenous substances and mineral salts, such as wine, cider, beer or malt extract. Numerous experiments have shown that a fluid containing from 4 to 6 per cent. of acetic acid and the same quantity of alcohol with the addition of a small quantity of one of the above-mentioned fluids is best adapted for the vigorous nourishment of the vinegar ferment. Ordinary table vinegar contains as a rule from 4 to 6 per cent. of acetic acid; the average percentage of alcohol is in wine from 8 to 10; in cider from 4 to 6; and in beer from 3 to 5. Taking this statement as a guide, the preparation of a fluid containing from 4 to 6 per cent. of acetic acid, 4 to 6 per cent. of alcohol, and the required nitrogenous combinations and salts will not be difficult.

Fluids of this composition are obtained by mixing, for in-

stance, equal parts of cider and vinegar, or one part of wine with two of vinegar, or one part of beer with three of vinegar, and adding 5 per cent. of 90 per cent. alcohol to the mixture. Such mixtures, possessing the power of vigorously nourishing the vinegar ferment, can at the same time be considered as types for the preparation of alcoholic liquid of suitable composition.

To assure the exclusive development of vinegar ferment upon any of the above-mentioned mixtures it is best to heat it to the boiling-point of water. Young wine as well as cider contains considerable quantities of albuminous substances in solution, and fluids of this nature being well adapted for the nutriment of the mold ferment, the development of the latter might increase to such an extent as entirely to suppress the vinegar ferment and thus render its cultivation a failure. Beer is also very suitable for the nutriment of the mold ferment, though in a less degree than young wine, and besides living yeast, contains alcoholic ferment.

By heating wine or beer only for a moment to about  $158^{\circ}$  F., a large portion of the albuminous substances in solution becomes insoluble, and on cooling separates as a flaky precipitate, all ferments present in the fluid being at the same time destroyed. Hence for the preparation of a fluid especially adapted for the cultivation of pure vinegar ferment, it is recommended quickly to heat to the boiling point 1 quart of ordinary white wine in a covered porcelain vessel, and after cooling to the ordinary temperature, to mix it with 2 quarts of vinegar. To remove the separated insoluble albuminous substances, filter through blotting paper.

To prepare a nourishing fluid from beer, heat a quart of it to the boiling point, mix it after cooling with 3 quarts of vinegar, add  $\frac{1}{5}$  quart of 90 per cent. alcohol, and filter.

Distribute this fluid in a number of shallow porcelain vessels and place the latter near a window in the heated workroom. To prevent dust from falling into the fluid, cover each dish with a glass plate resting upon two small wooden sticks placed

across the dish. In two or three days, and sometimes in 24 hours, the commencement of the development of the vinegar ferment will be recognized by the stronger odor of vinegar than that possessed by the original fluid and by the appearance of the surface of the liquid. By observing the latter at a very acute angle, dull patches resembling grease-stains and consisting of a large number of individuals of the vinegar ferment will be seen. In a few hours these patches will have increased considerably, until finally the entire surface appears covered by a very delicate veil-like layer of vinegar ferment.

By touching the surface with the point of a glass rod a certain amount of the coating adheres to it, and by rinsing it off in a fluid of similar composition not yet impregnated, the ferment quickly develops upon it. By placing a drop of the fluid under the microscope a picture similar to that shown in Fig. 2, p. 14, presents itself, the entire field of vision being covered with germs of vinegar ferment.

By the development of mold ferment the cultivation of pure vinegar ferment may sometimes result in failure even with the use of the above-mentioned fluids. The development of this ferment is recognized by the appearance of white dots upon the fluid, which quickly increase to white opaque flakes, and if left to themselves finally combine to a white skin of a peculiar wrinkled appearance. Fig. 35 shows a microscopical picture of such abortive culture of vinegar ferment. By observing at the commencement of this phenomenon the fluid with the microscope, very small individuals of vinegar ferment, *b*, will be observed alongside of the much larger oval cells *a*, of the mold ferment. Such fluid being not adapted for our purposes has to be thrown away and the dish rinsed off with boiling water.

When the fluid in the dishes is entirely covered with pure vinegar ferment, it is poured into a vessel containing the greater portion of the alcoholic fluid intended for the first charge of the generators, and in the course of 10 hours the entire surface of this fluid is covered with vinegar ferment.



This fluid being poured into the sufficiently acetified generators and trickling gradually through them, the greater portion of the ferment adheres to the shavings, and increases with such rapidity that the great rise of temperature in the interior of the generators shortly indicates the regular beginning of their activity, and the pouring in of alcoholic liquid can at once be commenced.

Vinegar ferment developed upon one of the above-mentioned fluids is evidently so constituted that it can be thoroughly

FIG. 35.



nourished by it, and hence the generators might be continued to be charged with alcoholic liquid of a corresponding composition. It being, however, as a rule, desired to manufacture as strong a product as possible, an alcoholic liquid much richer in alcohol than the above-mentioned nourishing fluids has to be used.

By, however, suddenly changing the nourishing fluid of the vinegar ferment, for instance, from a fluid containing only 4 to 6 per cent. of alcohol to one with 12 to 13 per cent., the action of the ferment would very likely be sluggish before it

became accustomed to the new conditions. Further, its activity might suffer serious disturbance and its propagation decrease very sensibly, so that notwithstanding strong heating of the workroom and thorough ventilation of the generators, the temperature in the latter would suddenly fall, and would only be restored to the required degree after the ferment had become accustomed to the new conditions and recommenced its vigorous propagation.

To overcome such annoying disturbances, it is only necessary to gradually change the composition of the nourishing fluid to that which the alcoholic liquid to be worked in the generators is to have. Commencing, for instance, with an alcoholic liquid containing 5 per cent. of alcohol, the next day one with 6 per cent. is used, the succeeding day one with 7 per cent., and so on until the maximum percentage of alcohol the liquid is to have is reached.

## CHAPTER XI.

### PREPARATION OF THE ALCOHOLIC LIQUID.

By the term "alcoholic liquid" is to be understood every kind of fluid to be converted into vinegar which, besides water and small quantities of nourishing salts and albuminous substance, does not contain over 14 per cent. of alcohol. The term "wash" or "mixture" is frequently applied to the alcoholic liquid. In the directions generally given for the preparation of such liquids, vinegar is mentioned as an indispensable constituent. While it cannot be denied that a content of vinegar in the alcoholic liquid exerts a favorable effect upon the formation of vinegar, it must be explicitly stated that it is not the acetic acid in the vinegar which in this case becomes active, but the ferment contained in it.

In a vinegar factory, vinegar just finished and quite turbid is as a rule used in the preparation of alcoholic liquid, and a

microscopical examination shows such vinegar to contain innumerable germs of vinegar ferment. This ferment on coming in contact with much air in the generators will evidently increase rapidly and contribute to the rapid acetification of the alcohol. That it is actually the ferment in the vinegar used which exerts a favorable effect can be shown by a simple experiment. By adding vinegar previously heated to from  $140^{\circ}$  to  $158^{\circ}$  F. to the alcoholic liquid, the formation of vinegar in the generators proceeds more slowly, the ferment contained in the vinegar having been killed.

The best proof, however, that the alcoholic liquid does not require any considerable quantity of acetic acid for its conversion into vinegar is furnished by the behavior of wine. Properly prepared wine of a normal composition contains only a few ten-thousandths of its weight of acetic acid, and this must very likely be considered as a product of vinous fermentation. If such wine be stored for years in a cool cellar, its content of acetic acid does not change. By, however, exposing it in a shallow vessel to the air at from  $66^{\circ}$  to  $78^{\circ}$  F., microscopical examination will show the development of vinegar ferment upon it, and a chemical analysis a constant increase, soon amounting to several per cent. of acetic acid. A fluid composed of 5 to 6 per cent. of alcohol, 94 to 95 per cent. of water, and a very small quantity of malt extract, acts in a similar manner. In many cases the vinegar ferment is developed without the fluid containing acetic acid.

The alcoholic fluid to be used may from the start contain a sufficiently large percentage of alcohol to correspond to the desired strength of the vinegar to be made, and in this case the fluid has to be poured several times into the generators, it being impossible to convert a large quantity of alcohol into acetic acid by passing it through but once. By another method an alcoholic liquid is first prepared containing but little alcohol, which is almost completely converted into acetic acid by one passage through the generators. The fluid running off from the generators is then further mixed with a cer-



tain quantity of alcohol, and being poured into a generator in which the vinegar ferment is already accustomed to larger quantities of alcohol and vinegar, is also converted into acetic acid. More alcohol can then be added, and so on. The last method is evidently the best as regards the conditions of life of the vinegar ferment, and actually the only one by which the strongest vinegar (with from 12 to 13 per cent. of acetic acid) can be obtained in generators.

That it is advisable only gradually to increase the content of alcohol in the alcoholic liquid is shown by the behavior of the ferment towards alcohol and acetic acid. Both bodies, if present in large quantities, are decidedly inimical to the propagation of the ferment, a fluid containing from 14 to 15 per cent. of alcohol, or as much acetic acid, being capable of checking it to such an extent as to disturb the process of production.

Another argument against the use of the total quantity of alcohol in the preparation of the alcoholic liquid to be employed for the first pouring, is the fact that evidently more alcohol will be lost by evaporation than by commencing with a fluid containing less alcohol, and adding a corresponding quantity of the latter after the fluid has once passed through the generators. The quantity of alcohol for the first pouring should be so chosen that the fluid running off still contains a small quantity of unchanged alcohol, this being an assurance that only alcohol and not unfinished acetic acid has undergone an alteration. So long as alcohol is present in the alcoholic liquid the vinegar ferment is almost entirely indifferent towards acetic acid, but after the oxidation of all the alcohol, it attacks the acetic acid and decomposes it to carbonic acid and water. This can be shown by a very simple experiment. If finished vinegar, instead of alcoholic liquid, be poured into a generator in full operation, the vinegar running off shows a smaller percentage of acetic acid than that poured in, the acetic acid missing having been destroyed by the ferment.

To what an extent even smaller quantities than 14 to 15 per cent. of alcohol or acetic acid exert a restraining influence

upon the propagation and activity of the vinegar ferment can be seen in generators charged with alcoholic liquid of different strengths, those containing less concentrated liquid can in the same time form a much larger quantity of acetic acid than those in which a liquid is used which already contains certain quantities of acetic acid. Hence, the greater the quantity of acetic acid already contained in the alcoholic liquid, the slower the conversion of the alcohol still present in the acetic acid.

It may, therefore, be laid down as a rule that the manufacturer should not strive to prepare vinegar with more than about 12 per cent. of acetic acid. Though in exceptional cases a product with 13 per cent. can be obtained, it will also be observed that the respective generators gradually yield a weaker product, or that their activity suddenly ceases to such an extent as to require them to be placed out of operation.

The preparation of high-graded vinegar being undoubtedly subject to greater difficulties than that of a weaker product, the question might be raised whether the manufacture of weak vinegar only would not be the most suitable. This must be largely decided by local conditions. For a manufacturer whose custom lies in the immediate neighborhood, for instance, in a large city, the production of weak vinegar only would be advisable; but if he has to send his product a considerable distance, the fact that the more freight has to be paid on what is of no value, the weaker the vinegar is, and that the expense of transporting a strong article is relatively less, deserves consideration. The consumer can readily prepare vinegar of any described strength by diluting the strong product with water.

The quantity of beer required for the purpose of offering suitable nutriment to the vinegar ferment is very small, an addition of 1 per cent. to the alcoholic liquid being ample. Sour or stale beer can of course be used. The reason for the employment of larger quantities of beer in mixing the alcoholic fluids is found in the fact that the vinegar prepared from such mixtures sooner acquires a pure taste than that made from fluids containing but little beer. The addition of beer should,

however, not exceed 15 per cent. of the total quantity of alcoholic liquid, as on account of the comparatively high percentage of albuminous substances and the maltose, dextrin, and extractive matters of hops it contains, a larger quantity would be injurious to the process of acetic fermentation, the generators being frequently rendered inactive by the so-called "sliming of the shavings." The production of the latter is due to the fact that by being partially excluded from contact with the air by the comparatively thick fluid passing over it, the vinegar ferment deposited upon the shavings assumes the form of mother of vinegar which adheres to the shavings as a slimy mass.

The quantity of finished vinegar added to the alcoholic liquid varies between 10 and 33 per cent. The use of large quantities is however decidedly inexpedient, since the only effect produced by the vinegar is, as previously stated, due to the ferment contained in it. Of freshly prepared, turbid vinegar, 10 per cent. is ample for the preparation of alcoholic liquid, and a greater quantity can only be considered as useless ballast.

Theoretically a certain quantity of alcohol yields exactly a certain quantity of acetic acid. The following table shows the proportions between the two bodies:—

A mixture containing the following per cent. of alcohol by volume	Is composed by weight of		And yields		Total vinegar.	Percentage of acetic anhydride.
	Alcohol.	Water.	Acetic anhydride.	Water.		
1	0.8	99.2	1 0	99.5	100.5	1.0
2	1.6	98.4	2.1	99.0	101.1	2.1
3	2.4	97.6	3.1	98.5	101.6	3.1
4	3.2	96.8	4.2	98.0	102.2	4.1
5	4.0	96.0	5.2	97.6	102.8	5.1
6	4.8	95.2	6.3	97.1	103.3	6.0
7	5.6	94.4	7.3	96 6	103.9	7.0
8	6.4	93.6	8.3	96.1	104.4	8 0
9	7.2	92.8	9.4	95.6	105.0	8.9
10	8.1	91.9	10.4	95 0	105.4	9.9
11	8.9	91.1	11.6	94.6	106 2	10.9
12	9.7	90.3	12.6	94.1	106.7	11.8



Practically less vinegar with a smaller percentage of acetic anhydride is, however, always obtained, this being due to losses of material caused partially by evaporation and partially by the oxidation of the alcohol extending beyond the formation of acetic acid. In preparing the alcoholic liquid these unavoidable losses must be taken into consideration, and more alcohol has to be used for the production of vinegar with a determined percentage of acetic acid than is theoretically required. How much more has to be taken depends on the kind of apparatus used and on the strength the vinegar to be prepared is to show. The higher the percentage of acetic acid which is to be obtained, the greater the losses will be, and consequently the greater the content of alcohol in the alcoholic liquid must be. Theoretically one per cent. of alcohol yields one per cent. of acetic acid, but practically the proportions are as follows :

For the production of vinegar with a con- tent of acetic acid of—	Is required an alcoholic liquid with a content of alcohol of—
5 per cent . . . . .	5.4 to 5.5 per cent.
6 “ . . . . .	6.5 “ 6.6 “
7 “ . . . . .	7.6 “ 7.7 “
8 “ . . . . .	8.7 “ 8.8 “
9 “ . . . . .	9.8 “ 9.9 “
10 “ . . . . .	10.9 “ 11.0 “
11 “ . . . . .	11.9 “ 12.1 “
12 “ . . . . .	13.0 “ 13.2 “

The strength of commercial alcohol varying considerably, it is of importance to the manufacturer to be able to calculate in a simple manner how many gallons of water have to be added to alcohol of known strength in order to obtain an alcoholic liquid with the desired percentage of alcohol. The calculation is executed as follows :—

Suppose :

P = per cent. of alcohol in the spirits to be used.

E = per cent. of alcohol in the alcoholic liquid to be prepared, the quotient obtained by dividing P by E gives the volume to which the spirits have to be reduced by the addi-

tion of water in order to obtain alcoholic liquid with the desired percentage of alcohol.

Example:—

From spirits of 86 per cent. Tralles', alcoholic liquid with 11 per cent. of alcohol is to be prepared.

$$P = 86 ; E = 11 \qquad \frac{P}{E} = 7.818.$$

Hence one volume of the spirits to be used has to be brought to 7.818 volumes, or to one gallon of spirits 6.818 gallons of water have to be added.

Examples of the composition of alcoholic liquid:—

A. Alcoholic liquid from alcohol, water, and vinegar:

*For vinegar with about 7 per cent. of acetic acid.*—Alcohol of 90 per cent. Tr. 10 parts by volume, water 107, vinegar with 7 per cent. of acetic acid 12.

*For vinegar with about 12 per cent. of acetic acid.*—Alcohol of 90 per cent. Tr. 10 parts by volume, water 65, vinegar with 12 per cent. of acetic acid 7.

It is advisable to add about 1 per cent. of the entire volume of beer to the above alcoholic liquids.

B. Alcoholic liquid from alcohol, water, vinegar, and beer.

*For vinegar with about 5 per cent. of acetic acid.*—Alcohol of 90 per cent. Tr. 10 parts by volume, water 107, vinegar with 5 per cent. of acetic acid 13, beer 14.

C. *For vinegar with about 8 per cent. of acetic acid.*—Alcohol of 90 per cent. Tr. 10 parts by volume, water 92, vinegar with 8 per cent. of acetic acid 10, beer 9.

In many factories it is customary not to determine the composition of the alcoholic liquid by calculation, but simply to work according to certain receipts. Vinegar of a certain percentage is obtained, but its strength cannot be predetermined with the same nicety as by calculating the percentage of alcohol in the alcoholic liquid by the above formula. The following may serve as examples of such receipts:—

D. Alcohol of 50 per cent. Tr. 100 quarts, water 600, vinegar 900.

E. Alcohol of 90 per cent. Tr. 100 quarts, water 1200, vinegar 300.

F. Alcohol of 90 per cent. Tr. 100 quarts, water 1350, vinegar 175, beer 175.

G. Alcohol of 90 per cent. Tr. 100 quarts, water 1400, vinegar 300, beer 100.

H. Alcohol of 80 per cent. Tr. 100 quarts, water, 850, beer 750.

I. Alcohol of 50 per cent. Tr. 100 quarts, water 100, beer 100.

The mixtures A, B and C are only given as examples of how alcoholic liquids which yield vinegar containing the desired percentage of acetic acid are prepared according to receipts. Though it may be very convenient for the manufacturer to work according to such receipts as are given under D to I, their use without a previous examination cannot be recommended. It is far better for the manufacturer to prepare the alcoholic liquid according to a receipt of his own, and not shrink from the slight labor it involves. He has then at least the assurance of obtaining vinegar with exactly the percentage of acetic acid desired, and is in the position to obtain an accurate view of the entire process of the operation.

In the United States low wine containing 12 to 15 per cent. by volume of alcohol is as a rule used for the preparation of the alcoholic liquid. Some manufacturers prepare the sacchariferous mash themselves, allow it to ferment by the addition of yeast, and then distil off to between 12 and 15 per cent. by volume. As the manufacture of yeast is frequently combined with that of vinegar, the distillates obtained from the fermented liquors are after skimming off the yeast utilized for vinegar manufacturing purposes.

Alcohol being the initial material in the preparation of alcoholic liquid, it is necessary to know exactly the per cent. by weight of alcohol it contains. With the assistance of the



tables at the end of this volume, the content of alcohol in spirits of wine can be readily determined by means of the alcoholometer and thermometer.

With the temperature of the spirits of wine at exactly 59° F., it suffices to determine its specific gravity by testing with an aerometer and to find the indicated figure in Table I (Hegner's alcohol table). The figure in the next horizontal column gives the per cent. by weight, and the next the per cent. by volume of alcohol contained in the spirits of wine examined. Tables II, III and IV give data relating to the proportion between the specific gravity and per cent. by weight and volume of spirits of wine of various concentration, as well as the decrease in volume by mixing with water. Table V shows the relation between the statements of Tralles' alcoholometer and a few others used in different places.

The specific gravity as well as the volume of spirits of wine varies with the temperature, and the statements of the aerometer for temperatures above the normal of 59° F. requires a corresponding correction, the execution of which is simplified by the use of Tables VI and VII. It being desirable, especially during the cold season of the year, to raise the temperature of the spirits of wine by mixing with water, Table VIII shows how much water has to be added in order to obtain from 105.6 quarts of spirits of wine of known strength, whiskey of any desired concentration.

In order to know exactly the yield of acetic acid which is obtained from a given quantity of alcohol, the acetic acid contained in the vinegar added must necessarily be taken into account as well as the alcohol in the beer, which is, of course, converted into acetic acid. It is best to make the content of alcohol in the alcoholic liquid so that it produces vinegar whose strength corresponds with that of the vinegar added. If, for instance, vinegar with 7 per cent. of acetic acid is used, alcohol of 7.6 to 7.7 per cent. by weight would have to be employed according to the table on page 109. The following compilation shows the manner of preparing alcoholic liquid according to rational principles.

Suppose vinegar with 7 per cent. acetic acid is to be prepared. There would be required—

Spirits of wine of 7.6 to 7.7 per cent. by weight.....	105.6	quarts.
Vinegar with 7 per cent. of acetic acid .....	10.56	“
Beer .....	10.56	“

Suppose the beer contains, for instance, exactly 3 per cent. by weight of alcohol, hence 10.58 ounces in 10.56 quarts. According to this, a result of 126.78 quarts of vinegar with exactly 7 per cent. of acetic acid could not be expected, since 10.56 quarts of the alcoholic liquid do not contain, as should be the case, 26.82 to 27.18 ounces of alcohol, but only 10.58 ounces. Hence actually to obtain vinegar with 7 per cent. of acetic acid a sufficient quantity of spirits of wine will have to be added to the alcoholic liquid to increase the content of alcohol by 16.22 to 16.57 ounces, or spirit of wine with more than 7.6 to 7.7 per cent. by weight will have to be used from the start.

It will, of course, be understood, that the data given above hold good only for the quality of the vinegar in reference to its content of acetic acid, the factor of the qualitative yield being left out of consideration. The material lost in the course of production amounts, as previously stated, to at least 15 per cent., and in determining the quality of the vinegar to be produced this circumstance has to be taken into consideration.

The content of acetic acid in vinegar can be determined with great ease and accuracy (up to  $\frac{1}{100}$  per cent.) by volumetric analysis, and from the result of such determination it can be readily seen how near the correct proportion of alcohol in the alcoholic liquid has been attained, and should the latter contain too little of it, it can be readily brought up to the determined percentage by the addition of some strong spirit of wine, or, if too much, by the addition of some water.

*Constitution of the Fundamental Materials used in the Preparation of Alcoholic Liquids.*—Spirits of wine, water, vinegar, and

in most cases beer, constitute the fundamental materials for the preparation of alcoholic liquids.

Any kind of wholesome drinking water is suitable for the manufacture of vinegar. Water containing a large amount of organic substance or living organisms, or which possesses a specific taste from the admixture of salts, should not be used under any circumstance.

Many well-waters are very hard, *i. e.*, they contain a comparatively large quantity of calcium carbonate in solution. If such water be used in the preparation of alcoholic liquid, the calcium carbonate is decomposed by the acetic acid and the vinegar contains a corresponding quantity of calcium acetate in solution. Other well-waters contain a large quantity of gypsum (calcium sulphate) in solution. This salt is not changed by acetic acid, but remains partially dissolved in the finished vinegar.

When water very rich in gypsum is mixed with alcohol the fluid, at first entirely clear, becomes in a short time opalescent and finally perceptibly turbid. After long standing a very delicate white sediment separates on the bottom of the vessel, the fluid becoming again clear. This phenomenon is explained by the fact that gypsum, while soluble in water with comparative ease, is next to insoluble in a fluid containing alcohol, and hence gradually separates in the form of minute crystals.

Water containing no gypsum but much calcium carbonate shows after mixing with spirits of wine a similar behavior; it at first becoming turbid and again clear after separating a delicate white precipitate. Calcium carbonate is soluble only in water containing a corresponding quantity of carbonic acid; on standing in the air the carbonic acid escapes and the calcium carbonate separates.

This behavior of water when mixed with alcohol and standing in the air can be utilized for the almost complete separation of the gypsum and calcium carbonate. Mixtures of water and alcohol, in the proportion the alcoholic liquids are to have, are



first prepared and the fluid stored in barrels in a warm apartment near the workroom. The mixtures at first turbid become clear after some time, and are then drawn off from the sediment by means of a rubber hose. A comparative examination of the water and the mixtures shows that the latter contain only very small quantities of gypsum and calcium carbonate in solution.

River water, though generally soft, *i. e.*, poor in the above-mentioned salts, is seldom sufficiently clear to be used without previous filtration. It is further very likely that the small worms, known as vinegar eels, which frequently become very annoying in vinegar factories, reach the alcoholic liquid through the use of river water, and, therefore, the use of well-water wherever possible is recommended.

The constitution of the spirits of wine used in the preparation of the alcoholic liquids is of great importance, the bouquet of the vinegar to be prepared depending on it. Commercial spirits of wine always contains certain foreign bodies known as "fusel oils," which have a very intense odor and can only be removed by careful rectification. For the vinegar manufacturer it is of great importance to know the behavior of spirits of wine containing fusel oil when converted into acetic acid, and a number of experiments with different varieties (from potatoes, grain, wine) have shown the respective vinegar also possessed of a specific odor, differing, however, from that of the original fusel oil and developing by storing into a bouquet of a peculiar but agreeable odor. This phenomenon is explained by the fact that the energetic oxidizing process which takes place in the generators extends not only to the alcohol but also to the other bodies present, and the greater portion of the fusel oils is thereby converted into odoriferous combinations or compound ethers.

By treating potato fusel oil (amyl alcohol) with sulphuric acid and an acetate, amyl acetate is formed which in a diluted state smells like jargonelle pears and is used by confectioners under the name of "pear essence" for flavoring so-called fruit

bonbons. The same process would seem to take place by passing spirits of wine containing potato fusel oil through the generators, the vinegar prepared from such spirits of wine showing an agreeable odor immediately when running off from the generators, while vinegar prepared from entirely pure spirits of wine has at first a stupefying smell and acquires a harmonious odor only by long storing.

It would, therefore, be advisable for the manufacturer who works with potato alcohol not to use the highly rectified product, but a mixture of it and of crude spirits containing fusel oil, the vinegar prepared from such a mixture acquiring a more agreeable odor than that obtained from the rectified product. How much of the crude spirits has to be used can only be determined by experience, but, as a rule, only enough should be taken to assure the conversion of the entire quantity of amyl alcohol present.

The fusel oil contained in spirits of wine from grain consists largely of a mixture of fatty acids, and offers far greater resistance to oxidation in the generators than amyl alcohol. The same may be said of œnanthic ether, the fusel oil of brandy. In working with alcoholic liquid prepared with a large quantity of grain spirits containing fusel oil, the smell of unchanged fusel oil is perceptible in the vinegar besides the odors of the products of its decomposition. With the use of small quantities of grain spirits containing fusel oil, vinegar possessing a more agreeable odor than that from entirely pure spirits is obtained.

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## CHAPTER XII.

### EXECUTION OF THE WORK IN A VINEGAR FACTORY.

WHEN the factory is in proper working order the further execution of the operation is very simple, it being only necessary to admit at stated intervals to the generators a previously

determined quantity of alcoholic liquid and to collect the vinegar running off. With the operation running its proper course, attention has only to be paid to the maintenance of the correct temperature in the workroom and in the generators, the chemical process proceeding regularly without further assistance. In many cases, however, deviations from the regular order occur, and are due to external influences, such as changes in the temperature in the generators, variations in the composition of the alcoholic liquid, etc. They will later on be discussed in a special chapter.

The capacity of a factory depends on the number of generators in operation. A regularly working generator is supposed to be capable of daily converting 3 liters (3.16 quarts) of absolute alcohol, and this quantity will be taken as the basis for calculating the execution of the operation. If, for instance, vinegar with 8 per cent. of acetic acid is to be manufactured, alcohol of 8.8 per cent. by weight has to be used, and to prepare this, 3 liters of 100 per cent. alcohol have to be reduced with water, so that, according to Table I, the fluid shows a specific gravity of 0.9858 at 59° F. According to Table III, 8.98 liters of water have to be added to every liter of 100 per cent. alcohol to obtain spirits of wine of 8.8 per cent. by weight; hence 3 liters have to be compounded with 26.94 liters of water (according to Table III, alcohol with 90 per cent. by volume of alcohol contains 11.80 per cent. by volume of water, 80 per cent. alcohol 22.83, etc., which has to be taken into consideration in making the dilution).

According to Table III, the contraction in this case amounts to 0.799 part by volume for every 100 parts by volume of the fluid. Hence the 3 liters of 100 per cent. alcohol yield, when diluted to spirits of wine of 8.8 per cent. by weight,  $26.94 + 3 = 29.94$  liters of fluid. Actually the quantity is somewhat smaller, as in mixing alcohol with water a decrease in volume takes place. If the alcoholic liquid is to contain 10 per cent. each of vinegar and beer, the quantity of fluid is as follows:—



Dilute spirits of wine . . . . .	, 29.94 litres.
Vinegar with 8 per cent. acetic acid . . . . .	2.994 "
Beer . . . . .	2.994 "
	35.928

Hence the quantity to be worked in a generator in the course of a day amounts to 35.928 liters, or taking into account the quantity of alcohol (about 90 grammes or 3.17 ozs.) contained in the beer, to about 36 liters. This quantity has to be divided among the separate pourings so that in a working time of 15 hours, 2.4 liters would have to be poured every hour. However, by this method, too much alcohol would, on the one hand, be lost by evaporation, and, on the other, the work of the generators would be comparatively slow, since, as is well known, the conversion into acetic acid is effected with greater rapidity when the alcoholic liquid contains less alcohol. Hence it is advisable to use in the commencement of the operation a fluid which contains only about one-half or two-thirds of the total quantity of alcohol, and to add a corresponding quantity of strong alcohol to every fresh pouring.

When all the alcohol has been converted into acetic acid, the vinegar ferment, as previously mentioned, commences, with great energy to oxidize the latter to carbonic acid and water, and hence the quantity of spirits of wine added to the alcoholic liquid must be sufficiently large for the *vinegar running off* to contain always a minute quantity of it.

Much has been written about this gradual strengthening of the alcoholic liquid with alcohol, and explicit directions are given as to the original composition of the alcoholic liquid, as well as to how much, how often, and when the alcohol is to be added. These directions may have proved useful in many cases, but local conditions exert too great an influence upon the process of manufacture for them to be of general value. Besides the content of alcohol in the alcoholic liquid, the size of the generators, the strength of the draught in them, the temperature prevailing in the workroom and in the interior of the generators, are factors which must be taken into consider-

ation in determining on a plan of operation actually adapted to existing conditions.

The size of the generators is, of course, fixed once for all. In a proper state of working the strength of the current of air must be so regulated that the temperature in the interior of the generators is *only about 45° F. higher than that of the work-room*, which is readily accomplished with a suitable central heating apparatus. There still remains the determination of the most favorable proportion of the content of alcohol in the alcoholic liquid to be first used and its gradual strengthening by the addition of spirits of wine, which can only be effected by a chemical examination of the fluid running off from the generators.

This chemical examination is restricted to the accurate determination of the quantity of acetic acid in the fluid and to that of the alcohol to 0.1 per cent. The determination of the acetic acid is effected by volumetric analysis, and with some experience requires four to five minutes for its execution. For the determination of the alcohol an examination with the ebullioscope suffices, which can also be accomplished in four to five minutes.\* These two determinations, which every vinegar manufacturer should be able to make, are the only means of obtaining an accurate control of the working of the factory, and also serve, of course, for settling the exact plan of operation from the start.

If, with reference to the example given above, vinegar with 8 per cent. of acetic acid is to be prepared, the alcoholic liquid must contain a total of 8.8 per cent. by weight of alcohol. Now if the manufacture is commenced with an alcoholic liquid containing the total quantity of water, vinegar, and beer, but, for instance, only 5 per cent. by weight of alcohol, the following method will have to be pursued in order to accurately determine when and how much alcohol has to be added.

The first portion of the alcoholic liquid being poured into

\* The manner of executing these determinations will be described later on.

the generator, the fluid running off is tested as to its content of acetic acid and alcohol, the test being repeated after the second and each successive pourings. Each test must show an increase in the content of acetic acid and a decrease in that of alcohol, and the latter must finally have disappeared so far that a new addition of alcohol seems to be in order. If the test after the third pouring shows the fluid to contain only 0.3 to 0.4 per cent. of alcohol, this quantity would be quickly and completely oxidized in the fourth pouring, and a certain quantity of acetic acid be at the same time destroyed. Hence it is necessary to add, for instance, 2 per cent. by weight of alcohol to the alcoholic liquid before the fourth pouring. When this  $2 + 0.3$  or  $2 + 0.4$  per cent. of alcohol, which the alcoholic liquid now contains, is again reduced, after the sixth or seventh pouring, to 0.3 or 0.4 per cent., the last addition of 1.8 per cent. of alcohol is made, the total quantity of alcohol,  $5 + 2 + 1.8 = 8.8$  per cent. having now been used.

When, after a certain number of pourings, a test of the fluid running off shows a content of 8 per cent. of acetic acid and only 0.1 or 0.2 per cent. of alcohol (a small remnant of alcohol should always be present) the process is considered as finished, and a further pouring into the generator would not only be useless labor, but contrary to the end in view, since, after the complete oxidation of the last remnants of alcohol, that of acetic acid would immediately commence, and weaker vinegar would be obtained after each pouring.

If a generator works up the quantity of alcoholic liquid intended for 12 or 15 hours in 10 or 12 hours, it is more proper, on account of the diminished loss by evaporation, to induce slower work by decreasing the draught of air in order to maintain the rule that a generator has to work up 3 liters of absolute alcohol in the working time of a day.

After controlling for several days the work of a generator, by examining the products as to their contents of acetic acid and alcohol, the plan of operation resolves itself from the results of these tests, since then it is accurately known after



how many pourings of an alcoholic liquid of known composition an addition of alcohol is required ; further, after how many pourings a finished product is present, so that directions for the progress of the operation can be given to the workmen according to time and quantities. The normal working of the generators can always be controlled by from time to time repeating the test of the products.

Now, suppose the work in a newly arranged factory having reached the point at which acetification is complete, the actual production, according to the old method, will be gradually commenced by pouring in alcoholic liquid of corresponding concentration.

The shavings of the generator having been saturated with acetifying vinegar, the latter is partially replaced by the fluid poured in, and as much as is expelled runs off. If the generator should at once commence to work regularly, the temperature in its interior would be observed to rise, though it would at first be impossible to establish a change in the composition of the fluid running off. Slight variations in the content of acetic acid and a small percentage of alcohol could be determined in the fluid only after the acetifying vinegar originally present has been entirely expelled by a series of pourings.

With the progress in the manufacture of vinegar, it became customary to produce the strongest vinegar possible, the so-called triple vinegar, with about 12 per cent. of acetic acid. On account of its greater commercial value, this article could be sent greater distances, the consumer reducing it to a weaker product by the addition of water.

To prepare directly vinegar with such a high percentage of acetic acid, it would, however, be necessary to acetify all the generators with vinegar of the same strength, and to use alcoholic liquid very rich in alcohol. By this method the losses of alcohol by evaporation, and also of acetic acid, would, however, be so great as to make the product too expensive. Furthermore, the work would require most careful and constant attention on account of the difficulty with which oxidation takes

place in alcoholic liquid containing much acetic acid, and it might only too readily happen that the generators suddenly worked with less vigor, *i. e.*, that the content of acetic acid in the vinegar running off would decrease, and the quantity of alcohol remaining unchanged correspondingly increase.

On account of these difficulties, it has become customary to charge the greater number of generators with alcoholic liquid yielding the so-called double vinegar with about 8 per cent. of acetic acid, and to work this vinegar with the addition of the required quantity of strong spirits of wine in a number of generators, which, of course, must be acetified with 12 per cent. vinegar.

It will be readily understood that the employment of this method is not only advantageous for the production of vinegar with the highest attainable content of acetic acid, but also for general purposes. Passing the alcoholic liquid but once through the generators does not suffice, even for vinegar with only 5 to 6 per cent. of acetic acid, an examination always showing a considerable quantity,  $\frac{1}{2}$  per cent. and more, of unconverted alcohol in the vinegar running off. The conversion of alcoholic liquid with a small content of alcohol into vinegar by one pouring can, to be sure, be accomplished, but it necessitates the use of very tall generators and a constant struggle with difficulties on account of the irregular draught of air, caused by the packing together of the shavings.

*Group System.*—Theoretically, as well as practically, the group system may be considered as the perfection of the quick process. The principle of the operation consists in the division of the generators into two or three groups, each group preparing vinegar of determined strength. In factories which do not produce vinegar of the greatest attainable strength (12 per cent. vinegar), but only double vinegar with about 8 per cent. of acetic acid, two groups might suffice. The manufacture of a product of the greatest attainable strength being, however, advisable in most cases, it is recommended to arrange the factory for continuous work with three groups of generators.

For this purpose the number of generators must be divisible by three; Hence 3, 6, 9, 12, etc., generators have to be provided, of which 1, 2, 3, 4, etc., form one group, so that, for instance, in a factory working with 24 generators belonging to one group with the same number, we have groups I, II and III, and in acetifying and operating, the generators belonging to one group are treated in the same manner.

For the preparation of the strongest vinegar (12 per cent.) the generators belonging to group I can, for instance, be acetified with vinegar of 6 per cent. acetic acid, those of group II with 9 per cent. vinegar, and those of group III with 12 per cent. vinegar. The process of operation is then as follows:—

Group I. The generators belonging to this group are charged with an alcoholic liquid which yields vinegar with a content of 6 per cent. acetic acid, and the fluid running off is poured back into the generators until a test shows the alcohol, with the exception of a small remnant, to have been converted into acetic acid. To this vinegar is then added sufficient strong alcohol to form an alcoholic liquid which will yield 9 per cent. vinegar.

Group II. The alcoholic liquid for 9 per cent. vinegar is poured into the generators belonging to group II, the pourings being repeated until all but a very small quantity of the alcohol is oxidized. The vinegar running off is again compounded with sufficient alcohol to form alcoholic liquid for 12 per cent. vinegar, and is brought into

Group III. The pourings are here repeated until the oxidation of alcohol is nearly complete. The finished product is then stored or clarified.

As will be seen from the above, in operating according to the group system, the entire factory is, so to say, divided into three factories, I, II, and III, of which I produces vinegar of 6 per cent., II vinegar of 9 per cent., and III vinegar of 12



per cent. The product of I, after having been converted by a suitable addition of alcohol into alcoholic liquid adapted for the preparation of 9 per cent. vinegar, is directly used for charging the generators of group II, and that of II for charging III.

The generators belonging to one group having been acetified with vinegar of the same strength, the fluid running off from one generator need not necessarily be returned to it. The work can, therefore, be simplified by conducting the fluid, running off from all the generators by means of a suitable pipe-system into a common receiver instead of allowing the fluid, which has passed through a generator, to collect under a false bottom and then drawing it off and returning it to the same generator. If, for instance, 8 generators belong to one group and 3 litres have at the same time been poured into each, the passage of the liquid through all the generators will be shown by a measuring scale placed in the common receiver, indicating that the latter contains  $3 \times 8 = 24$  litres.

The samples for determining the content of acetic acid and alcohol are taken from the common receiver, and the latter also serves for the conversion of the vinegar, after it has acquired the percentage of acid attainable in that group, into stronger alcoholic liquid by the addition of alcohol. In order to effect an intimate mixture, and at the same time prevent the vinegar ferment floating in the fluid from suffering injury by coming in contact with the highly concentrated spirits of wine, the required quantity of the latter is introduced in a *thin* jet and with constant stirring.

In many factories it is customary from time to time to alternate with the pourings in the groups or "to cross the generators." By this "crossing" the alcoholic liquid, which, according to the above method, would, for instance, pass from group II to group III, is poured into group I, so that after some time the generators of this group are converted into generators of group III (with 12 per cent. acid), and group III becomes group I, it now containing the weakest alcoholic liquid (with 6

per cent. acid). Crossing, however, cannot be recommended, because a sudden change in the constitution of the nourishing fluid always exerts an injurious influence upon the propagation of the vinegar ferment.

Recourse to crossing is most frequently had for the purpose of "strengthening" the vinegar ferment by working weaker alcoholic liquid in the generators of one group—generally that which yields the strongest vinegar—when their activity diminishes. This strengthening of the ferment can, however, be effected in a more simple and suitable manner by diminishing the quantity of alcoholic liquid poured in at one time and by increasing the draught of air, and the consequent change of temperature in the generators, so that the principal reasons for "crossing the generators" (which many manufacturers consider indispensable) have no force.

*Group System with Automatic Contrivances.* If the pourings of the alcoholic liquid are to be effected at determined intervals by an automatic contrivance, the group system as described on p. 122 *et seq.* should be used. The operation of such a factory is very simple. As seen from the description of the arrangement, the generators are divided into three groups, I, II, and III. Besides the generators each group must be provided with a reservoir, which may be designated *R*, and a collecting vessel *C*. (The other component parts, distributing arrangements and conduit, can here be left out of consideration.)

For the production of 12 per cent. vinegar in such a factory it is the best so to prepare the alcoholic liquid for the several groups that

Group I contains alcoholic					
liquid with	. . . .	6 p. c. acetic acid and 6.5 to 6.6 p. c. alcohol.			
Group II contains alcoholic					
liquid with	. . . .	9	"	"	+3.2 to 3.3
Group III contains alco-					
holic liquid with	. . .	12	"	"	+3.2 to 3.3

Group I having been acetified with 6 per cent. vinegar, group II with 9 per cent. vinegar, and group III with 12 per cent. vinegar, the fluid running off from group I, after being compounded with 3.2 to 3.3 per cent. of alcohol, is used in group II as alcoholic liquid for 9 per cent. vinegar, and yields 9 per cent. vinegar, which after being again compounded with 3.2 to 3.3 per cent. of alcohol, yields 12 per cent. vinegar after having passed through group III.

The uninterrupted working of the generators constituting one of the principal advantages of the automatic system, it is advisable to regulate the automatic contrivance so that but a small quantity of alcoholic liquid be at one time poured out, and to fix the intervals between two pourings so that the second pouring takes place after about one-half of the first has run off. Under these conditions there will be in the lower half of the generator an alcoholic liquid in which the alcohol is nearly as much oxidized as it can be by one passage through the generator, while in the upper half will be fresh alcoholic liquid in which oxidation is continued without interruption. A further advantage obtained by this is that a generator will yield quantitatively more than one working only 15 to 16 hours; further, the conditions of temperature in the interior of the generator remain always the same, and the ferment constantly finds nutriment.

The alcoholic liquid for group I is pumped into the reservoir  $R_1$ , and passes through the generators of group I into the collecting vessels  $C_1$ . All the alcoholic liquid having run off from  $R_1$ , the fluid collected in  $C_1$ , after having been tested as to its content of acetic acid, is for the second time pumped into  $R_1$  and passes again through the generators of group I. The automatic contrivance is so regulated that the alcoholic liquid, after being twice poured in, contains but a very small remnant of alcohol.

To the vinegar of 6 per cent. collected in  $C_1$  is now added 3.2 to 3.3 per cent. by weight of alcohol, best in the form of 80 to 90 per cent. spirits of wine. The resulting stronger alco-



holic liquid is at once pumped into  $R_2$ , and passing through the generators of group II reaches the collection vessel  $C_2$ . It is then tested, pumped back into  $R_2$ , and again collected in  $C_2$ . If it now shows the required strength, it is mixed with the second portion of 3.2 to 3.3 per cent. by weight of alcohol and is pumped into  $R_3$ , and after passing twice through the generators collects as finished vinegar in  $C_3$ .

It will be seen from the above description of the process that in making the tests, the product of all the generators of one group is treated as a whole. A disturbance may, however, occur in either one of the generators, and it would take considerable time before its existence would be detected by a change in the constitution of the entire product. The thermometer with which each generator is provided is, however, a reliable guide as to the activity of the latter, and if it shows in one of them a temperature varying from  $37^{\circ}$  to  $49^{\circ}$  F. from that prevailing in the others, it is a sure sign of the respective generator not working in the same manner as the others, and the product running off from it should be tested by itself as to its content of acetic acid and alcohol.

Generally it will contain either no alcohol or very much of it. In the first case the temperature of the respective generator is higher than that prevailing in the others, and its activity has to be moderated by decreasing the admission of air; in the other case, the generator works too sluggishly, and the difference is sought to be equalized by increasing the current of air or giving a few pourings of somewhat warmer alcoholic liquid. With a good heating apparatus producing a uniform temperature in the workroom such disturbances will, however, but seldom happen, and by the use of the above means the normal working of the generators can be restored.

## CHAPTER XIII.

## DISTURBING INFLUENCES IN THE MANUFACTURE OF VINEGAR.

IN no other industry based upon the process of fermentation are irregularities and disturbances of such frequent occurrence as in the manufacture of vinegar. Besides the nourishing substances dissolved in the fluid and free oxygen, the vinegar ferment requires a certain temperature for its abundant propagation, by which alone large quantities of alcohol can in a short time be converted into acetic acid. By exercising the necessary care for the fulfillment of these conditions serious disturbances can be entirely avoided, and the slighter ones due to insufficient acetic fermentation of the ferment readily removed.

As regards the nourishing substances of the ferment, irregularities can actually occur only in working continuously with an alcoholic liquid composed exclusively of water and alcohol. In such alcoholic liquid the nitrogenous substances necessary for the nutriment of the ferment are wanting, nor are the phosphates present in sufficient quantity. The consequences are the same as in every insufficiently nourished ferment-organism : The fermenting activity suddenly diminishes, propagation proceeds sluggishly and ceases entirely if abundant nutriment is not introduced. Hence it may happen that from a generator containing alcoholic liquid composed only of water, alcohol, and vinegar, the greater portion of the alcohol suddenly runs off unchanged, the temperature in the interior of the generator at the same time falling, and the draught of air ceasing soon afterwards. When these phenomena appear it should first be ascertained whether the disturbance is not due to too slight a current of air. For this purpose the draught-holes are entirely opened, and if the temperature rises the generator gradually resumes its normal working. If, however, no improvement is observed, the disturbance is due to defective nutriment, and the composition of the alcoholic liquid has to be changed, which is

best effected by the addition of a few per cent. of beer or of fermented alcoholic mash, either one of them containing a sufficient quantity of phosphates and albuminous substances. The use of sweet beer wort or malt extract has also been highly recommended for "strengthening weak-working generators." These substances also furnish albuminous bodies and phosphates to the alcoholic liquid, but they also contain maltose and dextrin, and as it has not yet been ascertained whether the latter and the carbohydrates in general can be consumed and digested by the ferment, they possibly may pass unchanged into the vinegar. Honey and glucose are also sometimes used for strengthening purposes, but while the former might be useful on account of the abundance of salts and nitrogenous substances it contains, no substances of any value to the ferment are present in the latter. At any rate the addition of beer, mash, or malt extract is to be preferred.

An addition of phosphate to the alcoholic liquid is also claimed to produce a favorable effect upon the propagation of the ferment. Commercial phosphoric acid is dissolved in water and the solution neutralized with potassium, a solution of potassium phosphate being in this manner obtained. The vinegar ferment being very sensitive towards this salt, a very small quantity of the solution, about  $\frac{1}{10000}$  of the weight of the alcoholic fluid may be added. The experiment must, however, be made very cautiously, and the effect upon the working of the generator carefully noted.

*Disturbances ascribable to the quantity of newly formed Acetic Acid.* Under proper working conditions the alcoholic liquid brought into the generators should be completely converted into vinegar, and theoretically, the product running off show the same strength as the vinegar used for acetification. Actually there are, however, slight variations not exceeding a few tenths of one per cent. Should greater differences appear, a disturbance actually exists and may show itself in various ways. The generator may work *too feebly* or *too vigorously*. In the first case the content of acetic acid in the fluid running off de-



creases considerably, while that of alcohol increases. The process of the formation of vinegar is, so to say, only half carried through, a great portion of the alcohol being converted, not into acetic acid, but into aldehyde. The greater portion of this combination is lost to the manufacturer on account of its low boiling point ( $71.6^{\circ}$  F.), it escaping in the form of vapor, the stupefying odor of which when noticed in the air of the workroom is accepted by all manufacturers as indicative of a disturbance in the regular working of the generators. This odor, however, becomes perceptible only after the disturbance has continued for some time, with the loss of a considerable quantity of alcohol. Hence the control of the working of the generators by a frequent determination of the acid becomes necessary. Repeated observations of the thermometer also furnish valuable hints about the progress of the chemical process. The temperature in this case remains only for a short time unchanged and soon falls, far less heat being liberated in the mere conversion of alcohol into aldehyde than when oxidation progresses to the formation of vinegar. These phenomena are indicative of the generator not being able to master the alcoholic liquid introduced, and may be due to the pourings being too large, or the temperature of the alcoholic liquid poured in being too low, or finally to an insufficient draught of air.

To restore the generator to a proper state of working, it is best to try first the effect of smaller pourings, and then an increased draught of air. If the disturbance was due to an insufficient draught of air, the temperature soon rises and the generator will be able to work up the regular quantity of alcoholic liquid. By the use of alcoholic liquid of a somewhat higher temperature the restoration of the normal conditions can be accelerated.

A decrease in the content of acetic acid in the fluid running off from the generators without the presence of alcohol being shown, indicates a too vigorous process of oxidation, the alcohol being not only oxidized to acetic acid, but the latter further

into carbonic acid and water. The temperature in the interior of the generators rises considerably, about 45° F., above that of the workroom.

In this case the restoration of the respective generator to a proper state of working is not difficult and can be effected in two ways, either by considerably decreasing the ventilation of the generator, or by pouring in a larger quantity of alcoholic liquid than previously used.

Heating of the generators is generally due to faulty construction. Generators of large dimensions, as a rule, become too warm much easier than smaller ones, the phenomenon also appearing more frequently in summer than in winter; and "too warm" being just as injurious to the efficacy of the generators as "too cool," they must, during the warm season of the year, be as carefully protected against too high a temperature as against cooling during the cold season. This is effected, on the one hand, by a suitable ventilation of the workroom during the night, and, on the other, by the use of alcoholic liquid of a somewhat lower temperature during the hottest season of the year. Moreover, disturbances from too high a temperature of the exterior air need only be feared in countries with a very warm climate.

It has been frequently proposed to counteract a too vigorous activity of the generators by the addition of a little oil of cloves or salicylic acid which have the property of checking fermentation. Salicylic acid, especially, is an excellent corrective for the faulty working of a generator. It has to be used, however, with great caution and only be added by the  $\frac{1}{100000}$  of the weight of the alcoholic liquid, and just in sufficient quantity to attain the desired result. A large amount is injurious to the ferment and might kill it.

*"Sliming" of the Shavings in Generators.*—This disturbance sometimes occurs in a vinegar plant, and its progress generally ends in throwing the entire operation into complete disorder so that finally no more vinegar can be produced. After fruitless experiments nothing remains but to empty the

generator, wash the shavings with hot water and, after drying and steeping them in hot vinegar, return them to the generator.

Sliming may be due to infection by foreign bacteria and fungi, as well as to super-oxidation and the accumulation of larger quantities of alcohol in the shavings which affects the bacteria to such an extent that they have no longer the power of forming acetic acid or only very small quantities of it, but only aldehyde, the intermediate product between alcohol and acetic acid.

The trouble begins to show itself by the generators commencing to work irregularly. While formerly a certain quantity of alcohol was after a fixed number of pourings converted into acetic acid, a large number of pourings are now required to attain the same result. The generators work slower and the heat in their interior decreases. By heating the workroom more strongly only a temporary improvement is brought about, and the production of the generators becomes less and less, and, finally, so low that work has to be interrupted. When the disturbance has progressed thus far a disagreeable musty, instead of the characteristic acid odor, is perceived in the workroom. By allowing one of the faulty working generators to stand for a few days without charging it with alcoholic liquid, the temperature in the interior may rise considerably and products of putrefaction be developed to such an extent as to taint the air of the workroom.

Long before this phenomenon becomes apparent, an alteration takes place in the shavings. A shaving taken from a normally working generator has the ordinary appearance of wet wood; but one taken from a generator working in the above-mentioned faulty manner is coated with a slimy mass, which is somewhat sticky, and can be drawn into short threads. Viewed under the microscope this slimy coating presents a structureless mass, throughout which numerous germs of vinegar ferment are distributed and sometimes the vinegar eels. Independently of the presence of the latter, this slimy coating



presents the same appearance as the so-called mother of vinegar. By placing a shaving coated with slime upright in a shallow dish, and filling the latter  $\frac{2}{3}$  the height of the shaving with alcoholic liquid, the previously described delicate veil of vinegar ferment develops upon the surface, while the portion of the shaving covered by the fluid is surrounded by flakes distinguished by nothing from mother of vinegar. Hence there can scarcely be a doubt that the slimy coating actually consists of the same structure to which the term mother of vinegar (see p. 21) has been applied, and in searching for the cause of the formation, it will generally be found to be due to conditions similar to those which give rise to the formation of the latter. An alcoholic liquid overly rich in young beer containing much albumen, or one to which much malt extract or young fruit-wine has been added, is apt to give rise to the formation of mother of vinegar in the generators. The slimy coating thus formed upon the shavings envelops the vinegar ferment and prevents its immediate contact with the air; consequently the alcoholic liquid does not encounter as much ferment as is required for the complete oxidation of the alcohol, and the generators become weaker. This decrease in the production is, of course, followed by a lower temperature in the generators, and consequently by a decrease in the propagation of the ferment, these unfavorable conditions finally becoming so great as to bring the activity of the generators to a standstill.

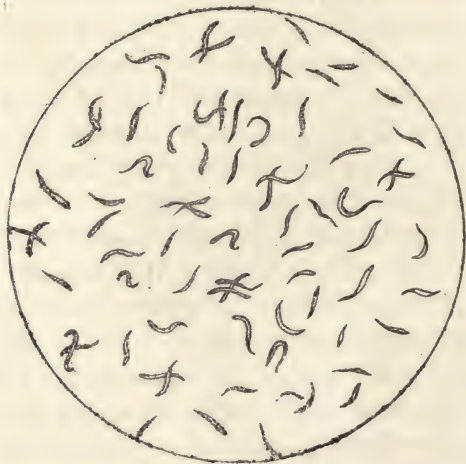
The settlement of vinegar eels upon the surface of the mother of vinegar has no connection with sliming. Should, however, large masses of these animalcules happen to die in the generators for want of air, due to the constantly decreasing draught, they quickly putrefy on account of the high temperature, and give rise to the most disagreeable odors.

A careful manufacturer will observe sliming at the commencement of the evil, when it can be remedied without much difficulty. First of all, the composition of the alcoholic liquid must be changed by discontinuing the use of fluid containing many carbohydrates and albuminous substances, such as

young beer, malt extract, young fruit-wine, it being best to use alcoholic liquid of water, vinegar, and alcohol only until the generators are entirely restored to a normal working condition. The activity of the ferment is at the same time increased by a stronger draught of air in the generators and by raising the temperature of the workroom. In a few days the generators will be again in a proper working condition, which is recognized by the normal conversion of alcohol into acetic acid.

If, however, the evil has progressed to a certain extent

FIG. 36.



nothing can be done but to empty the generators. Though considerable labor is connected with this operation, there is no further use of experimenting, since such nonsensical additions as beer-yeast, tartar, honey, etc., which have been proposed as remedies, only accelerate the final catastrophe—the entire cessation of the formation of vinegar. Should a disturbance occur which cannot be accounted for by defective nutriment of the ferment, want of air, or an incorrect state of the temperature, the condition of the shavings should be at once examined into, and if they show the first stages of sliming

the evil should, if possible, be remedied by changing the composition of the alcoholic liquid. If the new alcoholic liquid contains only water, vinegar, and alcohol, sliming cannot progress, and the layers of slime upon the shavings will in a short time disappear, they being partially utilized in the nutriment of the ferment, and partially mechanically washed off by the alcoholic liquid running down.

*Disturbances due to Vinegar Eels.* In many factories filamentous structures scarcely visible to the naked eye will frequently be observed in the vinegar. When viewed under the micro-

FIG. 37.



scope they will be recognized as animalcules, to which the term vinegar eel (*Anguilla aceti*) has been applied on account of their form slightly resembling that of an eel. Fig. 36 shows a microscopical picture of a drop of vinegar swarming with vinegar eels slightly magnified, and Fig. 37 a vinegar eel greatly magnified.

The animalcule consists of a cylindrical body running to a sharp point. The mouth-opening is covered with small knots; the throat is globular and passes directly into the long intestinal tube. The eggs are placed at about the centre of the



body in two tubes which unite to a plainly perceptible aperture. The average length of the female is 0.0682 Paris inch and that of the male 0.0486, the former being larger than the latter in proportion of 1 : 1.3.

Vinegar eels can exist in dilute alcohol of the strength used in making vinegar as well as in dilute acetic acid. In alcoholic liquid containing much alcohol and acetic acid they do not thrive as well as in weak liquid. Their part in the manufacture of vinegar is under all conditions an injurious one. The vinegar ferment can only carry on its function correctly when vegetating upon the surface of the fluid and in contact with air. The vinegar eel being an air breathing animal always seeks the surface, and in an alcoholic liquid which contains it, and upon whose surface an abundance of ferment grows, actual combats between animalcule and ferment can be observed, the former striving to force the latter, which is inimical to its existence, under the surface and thus render it harmless. (Submerged vinegar ferment, as is well known, changes its conditions of existence and becomes mother of vinegar.) If the conditions are favorable for the development of the animalcules, the latter overcome the ferment and submerge it so that it can continue to exist only as mother of vinegar, and consequently the process of the formation of vinegar will be considerably retarded. Under conditions favorable to the development of the ferment the reverse is the case. The ferment floating upon the fluid consumes nearly all the oxygen contained in the layer of air immediately above the surface, and thus deprives the animalcules of a condition necessary for their existence. A portion of them die and fall to the bottom of the vessel, while another portion of them escape to the sides of the vessel where they congregate immediately above the surface of the fluid in such masses as to form a whitish ring. These conditions can be readily induced by pouring vinegar containing a large number of vinegar eels into a flat glass dish and adding a fluid upon which vinegar ferment has been artificially cultivated. In a few hours the

ferment has spread over the entire surface and the animalcules form the above-mentioned white ring on the sides of the vessel. If by means of blotting paper the veil of ferment be removed as fast as it propagates, the animalcules soon spread over the entire fluid.

From the above explanation it is evident that the appearance of vinegar eels in large masses threatens danger to the regular working. When the animalcules reach the shavings the struggle for existence between them and the ferment commences, and their struggling to dislodge the latter may be the first cause of the formation of slimy masses of mother of vinegar upon the shavings. Since the vinegar eels consume oxygen, the air in the generators becomes thereby less suitable for the nourishment of the ferment, and consequently the generators will work feebly. By accelerating the draught of air in the generators, which is generally the first remedy tried, the development of the ferment may again become so vigorous that a large portion of the vinegar eels are killed, their bodies being found in the vinegar running off. The dead vinegar eels remaining in the generator, however, finally putrefy and give rise to the previously mentioned disagreeable odor. The processes of putrefaction being also effected by bacteria capable of decomposing nearly all known organic combinations (even small quantities of such strongly antiseptic bodies as salicylic and carbolic acids), it is evident that vinegar containing vinegar eels, cannot possess good keeping qualities and must be subjected to a special treatment, which will be referred to later on.

Several remedies for the suppression of vinegar eels in the generators have been proposed, one of them consisting of the introduction of vapors of burning sulphur, *i. e.*, sulphurous acid. Sulphurous acid, it is true, kills the vinegar eels, but at the same time, the vinegar ferment, and if small remnants remain, also the newly-introduced ferment. To restore a generator thus treated, a large quantity of air must be blown through it, which will remove the last traces of sulphurous acid. An alcoholic liquid containing much living ferment is then poured in.

The vinegar ferment can for many hours stand the exclusion of oxygen without being destroyed, while the vinegar eels die in a short time. This circumstance can be utilized for the destruction of the animalcules without recourse to other remedies. The generator having first been brought into the highest state of activity by pouring in very warm alcoholic liquid and opening all the draught-holes, is left to itself for 6 or 8 hours after closing all the draught-holes. The ferment in a short time consumes all the free oxygen in the generators, and the vinegar eels die from the want of it. By opening the draught-holes and pouring in alcoholic liquid, the normal formation of vinegar soon recommences.

The killing of a large number of vinegar eels in the above manner is, however, of considerable danger to the regular working of the factory, and the respective generators must be watched with special care in order to meet at once any appearance of putrefaction. It may sometimes succeed to keep up the work undisturbed, the killed vinegar eels being gradually removed from the generators by the vinegar running off. In such critical cases, when the generator may at any moment commence to work irregularly, the use of a very small quantity of salicylic acid as an addition to the alcoholic liquid would be advisable. The acid by checking putrefaction would prevent the immediate decomposition of the killed vinegar eels still present in the generators.

Should, however, signs of putrefaction appear, energetic means should at once be taken to arrest its progress, it being in this case best to sulphur the generator. This is effected by closing all the draught-holes except one, and introducing into the latter the nozzle of the apparatus whose arrangement is shown in Fig. 38.

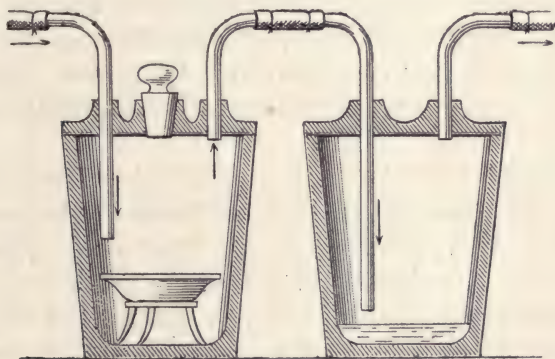
In a large clay vessel, best glazed inside, stands upon a tripod a shallow dish. The cover of the vessel luted air-tight with clay is provided with three openings. The opening in the center is closed by a well-fitting clay stopper, while glass tubes bent at a right angle and with a clear diameter of about  $\frac{1}{8}$  inch are



cemented in the openings at the side. The tube reaching nearly down to the plate is connected by means of a rubber hose with a double-acting bellows, while the second tube leading directly from the cover is connected with a second clay vessel. From the cover of this vessel a pipe leads to, and is fitted into, the open draught-hole of the generator.

For use the apparatus is put together, as shown in the illustration, and small pieces of sulphur are thrown through the central aperture upon the dish. The sulphur is ignited by throwing in a lighted sulphur match, and after closing the aperture the bellows is put in operation. The product of the

FIG. 38.



combustion of the sulphur passes through the tube into the generator, and in ascending dissolves the fluid adhering to the shavings to sulphurous acid. The addition of sulphur and the blowing-in of air are continued until the odor of burning sulphur is clearly perceptible in the upper portion of the generator. The second vessel which contains some water serves for the condensation of the portion of the sulphur which is not consumed, but only volatilized.

The sulphurous acid kills every living organism in the generator, and consequently *all the germs of the vinegar ferment are also destroyed.*

After allowing the sulphured generator to stand a few hours,

fresh air alone is forced through it by means of the bellows. The air-holes are then opened and the generator allowed to stand a few days for the sulphurous acid to be converted into sulphuric acid by the absorption of oxygen. To bring this generator again into operation, it is best to introduce at first a number of pourings consisting only of vinegar, with a content of acetic acid corresponding to that of the original acetification. In consequence of the absorption of sulphuric acid by the shavings, this vinegar becomes of no value as a commercial article, but it can be used for the preparation of alcoholic liquid.

The last traces of unchanged sulphurous acid having in this manner been removed from the generator and the greater portion of sulphuric acid adhering to the shavings washed out, the generator is again acetified, this being best effected by pouring in alcoholic liquid just run off from correctly working generators.

*Disturbances Due to Vinegar Lice (Vinegar Mites).*—Unless the most scrupulous cleanliness prevails, so-called vinegar lice will always be found in the factory. They prefer places kept constantly moist, and to which the air has access, for instance, the draught-holes and the interior of generators beneath the false bottom. As a rule, manufacturers do not pay much attention to their presence, as they apparently exert no influence upon the regular working. That such, however, is not the case, will be seen from the following occurrence: Some years ago, the proprietor of a vinegar factory in Italy informed Dr. Bersch, of Vienna, that millions of small animals had appeared in the factory and penetrated into the generators, the shavings to a certain height being covered with living and dead animals, and by reason of the latter putrefying, further operations had become impossible. Every drop of vinegar running off from the generators contained one or more of the mites. A small bottle half full of vinegar and closed air-tight by a cork accompanied the communication. Although the bottle had been sixty hours in transit, on opening

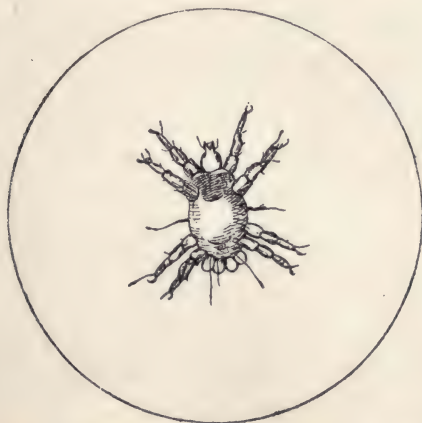
it a number of living animals were found, congregated especially in the fissures of the cork. On examining them with the microscope two forms (male and female?) could be clearly dis-

FIG. 39.



tinguished, many being only one-quarter or one-half the size of others. Figs. 39 and 40 show the two characteristic forms

FIG. 40.



of these animalcules. As far as it was possible to determine their zoölogical position, they belong to the family *Sarcoptidæ*.



No particulars as to their origin seem to be known, the manufacturer simply stating that they had come from the soil, under the supports of the generators and gradually rendered the latter ineffective. The generators were sulphured in the manner above described, and again put into operation.

To prevent the vinegar mites from collecting in large masses, scrupulous cleanliness must prevail in the factory. Especially should the draught-holes be from time to time examined, and, if mites be found, thoroughly cleansed with hot water, which kills them. The mites might also be prevented from penetrating into the interior of the generators by rings of a sticky substance (turpentine) around the draught-holes.

*Vinegar-Flies.*—Though, as far as known, the animals known as vinegar-flies create no disturbance in the regular working of the factory, they deserve mention because they appear wherever a fluid passes into acetic fermentation. In wine cellars, not kept thoroughly clean, these insects are frequently found on the bung-holes of the wine-barrels, and in factories in which the manufacture of wine vinegar is carried on according to the old system, they often occur in great swarms.

The vinegar-fly (*Drosophila funebris*, Meig) is at the utmost 0.11 inch long; it is especially distinguished by large red eyes sitting on both sides of the head and meeting in front. The thorax and legs are red; the abdomen, which is provided with six rings, is black, with yellow stripes. The wings are longer than the body. The larva is white, has twelve rings, on the mouth two black hook-like structures, and on the back part of the body four warts, two of which are yellow. In eight days the larva is transformed into a yellow chrysalis.

The collection of these flies in large masses can be readily prevented by keeping the factory thoroughly clean and being especially careful not to spill any fluid.

## CHAPTER XIV.

## SLOW PROCESS OF MAKING VINEGAR.

IN the manufacture by the slow process, barrels thoroughly cleansed with boiling water and previously saturated with hot vinegar are used. The bung-holes are left open or loosely covered. Smaller barrels with a capacity of from 15 to 25 gallons are preferred, and earthenware pots holding only 3 to 5 gallons are also used, it being claimed that they are especially suitable for the preparation of very strong vinegar. The barrels are arranged in tiers upon wooden supports in such a manner that their contents can be readily withdrawn by means of a faucet or a siphon. The heating apparatus may be either stoves, a hot-air furnace, or an arrangement similar to that employed in heating hot-houses. Due attention must be given to the methods of maintaining an equable temperature.

For the induction of the formation of vinegar, finished vinegar should be added to the dilute alcohol. By adding a few slices of bread, or beer wort, or a decoction of resins, the formation of vinegar can in many cases be accelerated, the substances named offering nutriment to the vinegar ferment.

The mixture of weak alcohol and vinegar is called *wash*. It is prepared from whiskey or alcohol, to which sufficient water is added that the mixture shows a content of about 6 per cent. of alcohol. To this weak spirit one-quarter or one-half of its volume of vinegar is added. Suppose vinegar containing  $4\frac{1}{2}$  per cent. of acetic anhydride is to be made. Theoretically, the wash should contain a little over 5 per cent. absolute alcohol, but on account of the loss by evaporation of alcohol, a wash of 6 per cent. must be used. If in making this wash 80 per cent. alcohol is employed, then the latter would have to be diluted so that every gallon of it becomes  $13\frac{3}{10}$  gallons. In other words, to 100 gallons of 80 per cent. alcohol 1,250 gallons of water are added, which makes 1,330 gallons of mixture, and

this, after the addition of 300 gallons of vinegar, becomes 1,630 gallons of wash. A portion of the water must be taken sufficiently hot to give a temperature of  $90^{\circ}$  to  $100^{\circ}$  F. to the wash. The resulting wash is placed in the fermenting barrels to fill each one two-thirds full, and the temperature of the apartment, observed by thermometers placed in different parts of it, must be kept at between  $75^{\circ}$  and  $100^{\circ}$  F. At the minimum temperature less fuel is required, but the time needed for acetification is extended, and consequently more barrels and a larger apartment are needed to make the same amount of vinegar. With the maximum temperature the reverse is the case.

Several days after the addition of the wash acetification begins, and is indicated by a temperature in the barrels slightly above that of the apartment. A piece of stone or slate, which is usually laid over the bung-hole of each barrel to prevent too great evaporation and consequent cooling, is bedewed with moisture, and a pungent acid odor is perceived in the room. As long as these indications continue, everything is going on well, but every barrel must be examined by itself to at once restore activity in any "lazy" one, lest putrefaction or mouldiness take place and spread to the neighboring barrels. When this misfortune occurs, the bad barrels are at once removed from the apartment, their contents thrown away, and the barrels scoured well with brushes and water, and placed in the sun. After they are dry they may be saturated with hot vinegar and brought into action again. If only "lazy," they are excited by withdrawing a portion of their contents, which is warmed in glass bottles, and with the addition of a little alcohol and vinegar is restored to the casks.

Too cool a location or a constant draught of air will sometimes put a cask out of action. This is remedied by removal, after acetification is restored, to a warmer location, or by covering with a non-conductor, such as heavy paper pasted over it.

After a lapse of time dependent on the temperature which



is kept somewhat higher towards the end of the operation, acetification is complete. Otto gives the time generally required as follows :

With a temperature of	Weeks required.
95° to 100° F.	2 to 4.
86° to 95° F.	4 to 8.
72° to 86° F.	8 to 16.

The close of acetification is indicated by the diminution of the strong vinegar smell in the room, by the absence of vapor condensing upon the slate covers of the bung-holes, and by the temperature of the inside of the barrels becoming equal to that of the room.

As soon as acetification in any one barrel is perfected, the vinegar must at once be withdrawn, barreled and removed to a cooler place than the vinegar room, in which its tendency to spoil in the heated atmosphere is very great. The slimy deposit called "mother of vinegar" is removed, and the vinegar with which it is imbued, employed in part for the next acetification. If the sediment from each barrel be placed in a cask, the clear vinegar may be drawn off after the deposition of the mother of vinegar. It is well before barreling the vinegar, to allow it to stand for a short time in a cool room in a vessel filled with beech shavings, which clarify it. When stored, a pint of spirits should be added to each barrel.

As previously mentioned, the slow process above described may be modified in various ways. Thus, instead of bringing the fermentation to completion in all of the barrels at about the same time, they may be divided into three or four groups, so that  $\frac{1}{3}$  or  $\frac{1}{4}$  of the whole quantity of vinegar may be withdrawn, and stored at intervals of  $\frac{1}{3}$  to  $\frac{1}{4}$  the time required for the acetification of the whole quantity. This modification has the advantage of a greater distribution of the work ; necessity of a smaller quantity of vinegar stored for sale, and the presence of barrels in full action, emitting strongly acetic vapors, which is of advantage in keeping up fermentation in barrels

just going into operation. The disadvantages consist in greater need for entering and leaving the vinegar room, involving loss of its heat, and requiring in consequence greater attention to its fires. In addition to this the heat cannot be increased towards the close of acetification, which is useful in shortening the time for manufacture.

Another modification consists in always keeping a large quantity of vinegar in the fermenting barrels, and at short intervals withdrawing small quantities of vinegar which are replaced by fresh wash. This saves time, as acetification is more rapid in the presence of large bodies of vinegar. It involves loss of heat by a need for too frequently entering the vinegar room. It involves also a loss of interest upon the value of the large quantity of vinegar kept in the fermenting barrels. The intervals at which vinegar may be withdrawn are closer in proportion to the heat of the apartment, which bears a ratio to the amount of fuel consumed.

By this method only  $\frac{1}{5}$  of the vinegar is removed at one time from each barrel; in other words, at intervals of one to two weeks, according to temperature, one gallon of vinegar is withdrawn from every 5 gallons in the fermenting barrels, and in its stead a gallon of wash is added. In a large factory, the latter process requires a large number of barrels of vinegar to commence operations. The vinegar must be either purchased or made gradually in the fermenting barrels, not withdrawing any until the barrels are sufficiently full. The advantage consists in a smaller number of fermenting barrels being required than by the method first described. Dr. Otto gives the following calculation for the number of fermenting barrels required for the slow process:

Suppose that it is required to furnish a barrel of vinegar per day, excluding Sundays, which would equal 312 forty-gallon barrels per year, the fermenting barrels would have each a capacity of  $\frac{1}{2}$  barrel, and since they are not filled with wash, and on account of unavoidable loss, four such barrels may be allowed to each barrel of vinegar made. What is added to make the

wash is, of course, not accounted as manufactured vinegar, as a like quantity must be added in the subsequent wash. From every four fermenting barrels, one barrel of vinegar may be sold, and hence 6 barrels of vinegar will require 24 fermenting barrels. If the workroom be so heated that the operation is completed in four weeks, 24 barrels of vinegar will have to be drawn off, to do which 96 fermenting barrels will be required. If, however, a lower temperature be maintained in the workroom, say to complete the process in 16 weeks, 4 times 96 = 384 fermenting barrels, will be required. In the latter case the expense of fuel is lessened, but that of the fermenting barrels is increased. Besides, a larger apartment will be necessary, which will involve a higher rent and greater expense for fuel in heating it. If the process be modified, as described, so that a large body of vinegar is always kept in the fermenting barrels, their number may, as before stated, be proportionately decreased.

This calculation affords the very best illustration of the superiority of the modern quick process, over the old slow method. To make one barrel per day by the quick process, a small room and two generators are the only requisites.

*Household Manufacture of Vinegar.*—The following method is to be recommended as simple, expedient, and furnishing a constant supply of vinegar with scarcely any trouble and at trifling cost:

Procure two barrels, the one for making, the other for storing the vinegar, barrels from which good vinegar has just been drawn being preferable. The storage barrel is always kept in the cellar, and the generating barrel in the house or cellar, according to the season. At the top of one of the heads of the storage barrel a small hole is bored for the circulation of air. The barrels lie on their side, and each of them is furnished with a wooden faucet. Their capacity is, of course, regulated by the yearly demand.

Suppose that the generator, filled to the level of the ventilating hole, contains 10 gallons, the manufacture will then



be carried on as follows: Seven gallons of vinegar of a good quality are placed in the barrel, and three gallons of warm alcoholic liquid are added. This alcoholic liquid is made as follows: If common 50 per cent. whisky be employed, have a small measure of 3 pints and a large one (a bucket) of 3 gallons. If 86 per cent. alcohol is used, let the small measure be for 2 pints. Put a small measureful of spirits in the large measure; fill quickly to the mark with boiling water, and pour by means of a funnel into the generator. Every two or three weeks, three gallons of vinegar are withdrawn from the generator and added to the storage barrel, and three gallons of alcoholic liquid are placed in the generating barrel as before.

Another method of working the casks consists in half filling the generator with vinegar, and adding every week so much of the alcoholic liquid that it fills the barrel in from 8 to 16 weeks, according to the season. Half the vinegar is then added to the storage cask, and the process then recommenced in the generator. The warmer the season the more rapid may be the manufacture.

*Preparation of Vinegar with the Assistance of Platinum Black.*  
—In considering the theory of the formation of vinegar it was mentioned that platinum in a finely divided state possesses the property of converting alcohol into acetic acid. This property of platinum has been utilized for the purpose of manufacturing acetic acid on a large scale. The apparatus used for this purpose consists of a small glass house, provided in the interior with a number of compartments. The shelves forming these compartments support a number of porcelain capsules. The alcohol to be acetified is poured into these capsules, in each of which is placed a tripod, also of porcelain, supporting a watch-glass containing platinum black or spongy platinum. In the roof and at the bottom of the apparatus are ventilators, so constructed as to admit of regulating the access of air. By means of a small steam-pipe the interior of the house is heated to 79° F. By this means the alcohol is gently evaporated, and coming in contact with the platinum black or sponge is acetified.

So long as the ventilation is maintained, the platinum black retains its property of oxidizing the alcohol. With an apparatus of 52 cubic yards' capacity and with 37 pounds of platinum black, 150 quarts of alcohol can daily be converted into pure vinegar. The drawbacks to this process are high prices for alcohol, and the large quantity of the very expensive platinum required for working on a manufacturing scale.

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## CHAPTER XV.

### FURTHER TREATMENT OF FRESHLY-PREPARED VINEGAR.

THE vinegar running off from the generators is "finished" in so far that it contains the quantity of acetic acid obtainable from the content of alcohol in the alcoholic liquid, but it becomes a commercial article only by long storing and special treatment.

The odor of freshly prepared vinegar is by no means agreeable. It is very pungent and at the same time stupefying, the latter property being no doubt due to small quantities of aldehyde contained in it, which, however, volatilize or oxidize by storing. The odor depends largely on the materials used in the manufacture, that of vinegar prepared from an alcoholic liquid composed of water and alcohol alone without an addition of beer being decidedly the least agreeable. By long storing such vinegar acquires a somewhat finer odor, but never especially agreeable properties.

The barrels for storing fresh vinegar should be filled up to the bung-holes and closed air-tight, since when air is present the ferment in the absence of alcohol consumes acetic acid, thus reducing the strength of the vinegar; and moreover, mold ferment might develop.

The temperature of the vinegar running off from the generators being quite high, its volume diminishes on cooling, and

consequently the barrels when inspected later on will not be quite full. When the vinegar is stored in barrels not made air-tight by a suitable coating (lacquer, paraffin, etc.), the air penetrates through the pores of the wood and a constant reciprocal action takes place between it and the vinegar. The very slow oxidation thus produced exerts a decidedly favorable influence upon the odor of the vinegar, the processes thereby taking place being somewhat similar to those which cause the formation of the bouquet in wine. This similarity extends also to the fact that the vinegar bouquet, if it may so be called, is the finer the slower the effect of the oxygen, and this can be reached by storing the barrels in a warehouse having a temperature of from 57° to 60° F., or in a cellar.

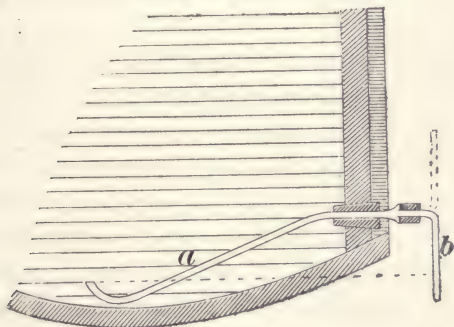
It has been sought to improve the odor of vinegar by various additions, but that of volatile oils, such as oils of caraway, fennel, anise, etc., which has been frequently proposed for the purpose, cannot be recommended. These oils, to be sure, give a specific, agreeable odor to the vinegar, but an expert can at once detect such additions. More suitable for the purpose is the use of a very small quantity (a few hundred-thousandths of the weight of the vinegar) of potato or grain fusel oil, these bodies forming with the corresponding quantity of acetic acid the frequently mentioned odoriferous compound ethers. An addition of  $\frac{1}{4}$  per cent. of very strong alcohol to the vinegar has also a very favorable effect upon the odor of the latter, acetic ether being formed in storing. In place of alcohol, acetic ether or amyl acetate (pear essence) can be directly added, but only in very small quantities and best in alcoholic solutions of a determined content, for instance, 50 grammes of pear essence to 1 liter of 95 per cent. alcohol. Of this solution 0.1 liter (= 100 cubic centimeters) contains 5 grammes of pear essence, and if added to 100 liters of vinegar, which in round numbers weigh 100 kilogrammes, the latter will contain  $\frac{5}{100000}$  of pear essence. By proceeding in this manner the correct quantity required can be readily determined. Immediately after the addition of one of the above-mentioned substances



its odor is disagreeably prominent, but becomes pleasant by storing.

After lying for several weeks a muddy sediment forms on the deepest place of the barrel. The vinegar can be carefully drawn off from this sediment by means of a rubber hose; or a special apparatus, similar to that shown in Fig. 41, is used for the purpose. It consists of the glass tube *a*, which is inserted in the tap-hole of the barrel and reaches to the bottom, where it is slightly bent upwards. In front of the bung-hole this tube is provided with a bulb in which is fitted by means of a cork a tube, *b*, bent at a right angle. While the vinegar is stored, this tube stands upright as indicated by the dotted

FIG. 41.



lines, and is secured to a rubber hose reaching up to the bung-hole. By turning the tube downward, the fluid runs out through the tube *a*, until its level has sunk to the dotted line.

Sometimes the vinegar is not rendered perfectly clear by storing, and filtering has to be resorted to. Before referring to this operation a few words will be said in regard to the storing of vinegar.

The vinegar brought into the storage barrels contains the following constituents: Water, acetic acid, alcohol (very little), aldehyde (very little), acetic ether, vinegar ferment (living and dead), extractive substances (depending on the

nature of the alcoholic liquid used). Moreover, there are frequently found alcoholic ferment (from the beer), and vinegar eels and vinegar mites, if these animals exist in the factory.

By filling the storage barrels to the bung-holes and closing them air-tight, the vinegar eels and vinegar mites die in a short time for want of air, and fall to the bottom. The living vinegar ferment present in the fluid must assume the form in which it can for some time exist without free oxygen, *i. e.*, of mother of vinegar. When in consequence of the shrinkage in the volume of the vinegar by cooling, the air penetrates through the pores of the wood, it is first utilized for the conversion of the small quantity of aldehyde into acetic acid, and later on enables the vinegar ferment to continue to exist upon the surface and to slowly convert the small quantity of alcohol still present into acetic acid.

If the barrels are not closed absolutely air-tight, the vinegar ferment will develop quite vigorously upon the surface, and when all the alcohol is consumed attack the acetic acid, so that when the vinegar is tested a decrease in the content of acetic acid is plainly perceptible. If the finished vinegar still contains considerable quantities of albuminous substances in solution (vinegar from grain, malt, or fruit), or if it contains tartaric and malic acids and at the same time only a small percentage of acetic acid, as most fruit vinegars do (seldom more than 5 per cent.), the mold ferment readily settles upon the vinegar and finally dislodges the vinegar ferment from the surface. The acetic acid is, however, very rapidly destroyed by the mold ferment, and through a luxuriant growth of the latter, which floats upon the surface as a white membranous coating, the vinegar may in a few weeks lose one or more per cent. of it. This happens so frequently, for instance with fruit-vinegar, that the opinion that such vinegar cannot be made to keep, is quite general.

Vinegar which, besides a considerable quantity of extractive substances, contains the salts of certain organic acids (malic and tartaric acids), for instance, vinegar prepared from apples

or wine, must be frequently examined, as it readily spoils, and may suffer even if kept in barrels constantly filled up to the bung. In fluids containing the salts of the above-mentioned organic acids a ferment may frequently develop, even when the air is excluded, which first decomposes the tartaric and malic acids, and though these acids are present only in a comparatively small quantity, they influence, to a considerable extent, the flavor of the vinegar on account of their agreeable acid taste. In vinegar in which this ferment has long existed a diminution of acidity can be readily detected by the taste, and by the direct determination of the acid a decrease in its content can be shown which, if calculated as acetic acid, may in some cases amount to one per cent. Besides the loss of its former agreeable taste, vinegar thus changed acquires a harsh tang, due no doubt to the formation of certain products not yet known formed by the ferment effecting the destruction of the tartaric and malic acids. Moreover, wine or fruit-vinegars in which this ferment has for a considerable time flourished, lose their characteristic agreeable bouquet which may be considered the greatest damage.

In the presence of a large number of vinegar eels their bodies may decay and impart to the vinegar a very disagreeable putrid odor, even if stored in barrels closed air-tight.

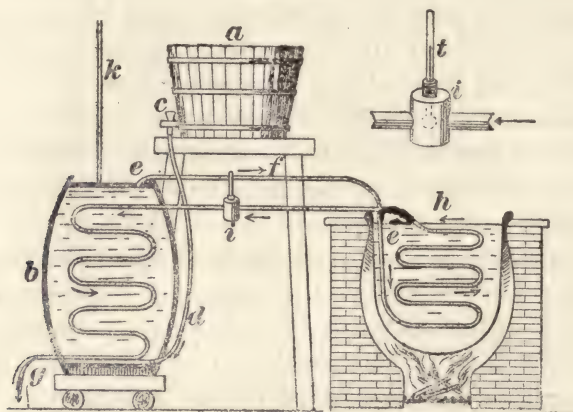
The advisability of filtering the vinegar before bringing it into the storage barrels will be readily understood from the above statement. By filtration it is, however, only possible to remove the vinegar eels and vinegar mites swimming in the fluid and larger flakes of mother of vinegar. The ferments and bacteria inducing putrefaction cannot be thus removed, so that even filtered vinegar is liable to spoil when stored.

*Heating the Vinegar.*—In order to destroy all organisms which might cause the spoiling of the vinegar, it is recommended to heat the latter to about 140° F. before running it into the storage barrels. A few moments exposure at this temperature being sufficient for the purpose, a large volume of vinegar can in a short time be heated with the use of a suitable apparatus, such as is shown in Fig. 42.



In the head of the barrel *b* is secured a pipe of *pure tin* with very thin walls and a clear diameter of about  $\frac{1}{8}$  inch. It is coiled in a boiler filled with water, which it enters at *e f* and leaves at *h*. It then passes into the barrel *b*, in which it is also coiled, and ends outside the barrel at *g*. At *i* it expands to a bulb in which a thermometer, *t*, is placed. A vat, *a*, placed at a certain height above the barrel is provided with a wooden stop-cock, *c*, to which is secured a rubber hose, *d*, which enters the barrel *b* above the bottom. The pipe *k*, which is secured on top of the barrel *b*, is open on both ends and of sufficient length to project above the vat *a*.

FIG. 42.



The boiler is filled with water and placed in an ordinary hearth. The vat *a* is filled with the vinegar to be heated and kept constantly supplied. The water being heated to boiling, the stop-cock *c* is opened. The vinegar now runs through *d* into the barrel *b*, and, after filling it, flows at *e* into the tin coil and in passing through it in the direction of the arrows is heated. The thermometer *t* dipping into the hot vinegar indicates the temperature, and the inflow of vinegar is accordingly regulated by opening or closing the cock *c*. As shown in the illustration, the hot vinegar runs through the coil sur-

rounded by cold vinegar into the barrel *b*, whereby it is cooled off and the vinegar in the barrel preparatorily heated. The pipe *k*, open on both ends, allows the escape of the gases developed.

In consequence of the albuminous substances becoming insoluble by heating, the vinegar running off at *g* is, as a rule, more turbid than before. It is brought into the storage barrels, which need, however, not be closed air-tight, the subsequent processes taking place in the vinegar being of a purely chemical nature and not caused by organisms. The latter have been killed by heating, and, together with all other foreign bodies suspended in the vinegar, gradually fall to the bottom of the barrel. If the vinegar after heating is allowed to lie for a sufficiently long time, it clarifies completely and can be drawn off perfectly bright from the sediment.

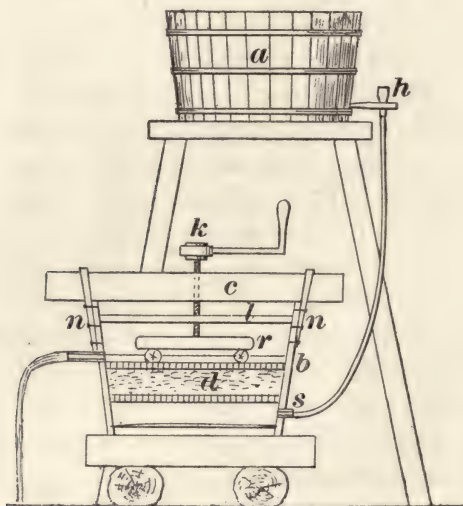
*Filtration of Vinegar.*—The bodies suspended in the vinegar and causing its turbidity being very small, it takes some time before they settle on the bottom and the fluid becomes entirely clear. To accelerate clarification the vinegar is filtered.

Fig. 43 shows a filter suitable for the purpose. It consists of a small, strong wooden vat provided with two perforated false-bottoms, *s* and *b*. Upon the lower false bottom is spread a linen cloth and upon it fine sand, which is not attacked by acetic acid, or small pieces of charcoal. Upon the smoothed surface of the sand is spread a layer of paper pulp  $\frac{3}{4}$  to 1 inch deep which is covered with a linen cloth and then placed upon the false bottom *b*, the latter being forced down by means of the screw *k* and the pieces of wood *r*. The vinegar to be filtered is in the vat *a* which is connected with the filtering vat by the stop-cock *h* and the rubber hose *s*, 8 to 10 feet long. By opening the stop-cock *h* the filter stands under the pressure of a column of fluid 8 to 10 feet high and the filtered vinegar runs off through an aperture in the side of the filtering vat. By filling the filter below the paper pulp with fine sand, the latter retains the greater portion of the solid bodies suspended in the vinegar, and it will be a considerable time

before the pores of the paper pulp become choked up to such an extent as to require its renewal.

Sharp, fine-grained sand should be used for filtering. It should be free from iron and sulphur and previous to use freed as much as possible from lime and earthy constituents by washing in pure water to which some hydrochloric or tartaric acid may be added. Fine white quartz sand is very suitable for the purpose. White sea-sand is also highly recommended for filtering vinegar, it being claimed that after

FIG. 43.



its use for several months not a single vinegar-eel was found in the filtered product. When after long use the sand becomes so closely packed that the vinegar does not run off with the rapidity desired, the layer of slime that has accumulated upon the sand is carefully removed and the sand thoroughly washed and dried, when it is again ready for use.

An arrangement suitable for filtering larger quantities of fluid under an increased pressure is shown in Fig. 44.

It consists of a strong linen bag, *S*, about 16 inches in diam-

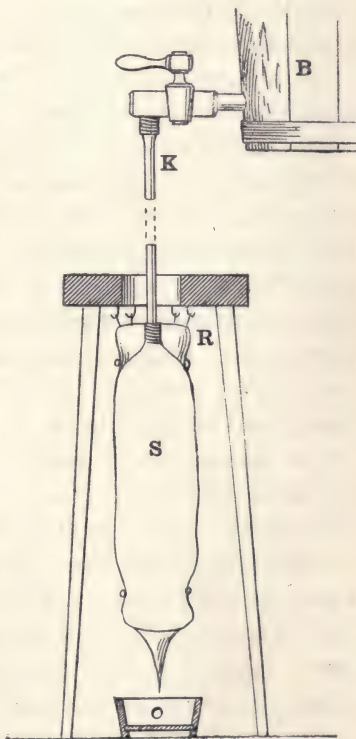


eter, and a jute or hemp hose, *R*, open at both ends and about 6 inches in diameter. The bag is tied by means of pack-thread to a cylindrical piece of wood which is secured to a suitable support. The bag is then connected by means of the rubber hose *K* with the reservoir *B*, which contains the vinegar to be filtered, and is placed about 10 to 13 feet above the support carrying the bag. The bag is folded so that it can be inserted in the hose *R*, the latter being also secured to the cylindrical piece of wood.

By gradually opening the stop-cock on the reservoir the bag is filled with vinegar, but being enveloped by the hose *R* cannot entirely expand but only so far as permitted by the diameter of *R*, so that though its entire surface acts as a filter a large number of folds are formed, and it is thus protected from bursting, even under the pressure of a column of fluid of considerable height. The fluid filtering through the bag runs down on the hose and collects in a vessel placed under it.

At first this filter generally does not act entirely satisfactorily, the fluid running off turbid; and this continues until the pores of the filter have become sufficiently contracted to retain the small bodies suspended in the fluid. This can, however, be remedied by stirring some charcoal powder into the first portion of vinegar to be filtered. The charcoal powder adheres to the sides of the

FIG. 44.



bag and contracts the pores of the tissue so that the fluid runs off entirely clear.

By subjecting the freshly-prepared vinegar to heating and filtering, a commercial article is obtained which is perfectly clear and does not spoil by keeping. By storing it, however, for some time in barrels it gains considerable in fineness of odor and taste. Wine-vinegar, cider-vinegar and fruit-vinegars in general should positively be stored for some time, the odoriferous bodies which make these varieties so valuable developing only by that means.

*Sulphuring of Vinegar.*—Sulphuring has long been employed as the most convenient method for the preservation of wine, and, if correctly applied, can also be used for that of vinegar. But as sulphurous acid readily dissolves in vinegar, the latter must not be brought in direct contact with the gases arising from the burning sulphur.

The sulphuring of vinegar is best executed as follows: The barrel intended for the reception of the vinegar is thoroughly rinsed and immediately placed in the storeroom. Then place a sulphur match consisting of a strip of linen about 6 inches long and  $\frac{3}{4}$  to 1 inch broad dipped in melted sulphur into a perforated sheet-iron cylinder about 8 inches long and 1 inch in diameter, secure this cylinder to a wire, and after igniting the sulphur match, lower it from the bung-hole to the center of the barrel. The sulphurous acid formed by the combustion of the sulphur is at once dissolved by the water adhering to the interior of the barrel. A sulphur match of the above size suffices for a barrel of 100 to 125 gallons.

If the sulphured barrel be now immediately filled with vinegar, the sulphurous acid becomes distributed throughout the fluid and kills the vinegar ferment as well as all other ferments present, so that the vinegar cannot undergo any further change except it come again in contact with living ferments.

The sulphurous acid dissolved in the vinegar is after some time converted into sulphuric acid and its presence can be readily detected. It may, however, be remarked that the

quantity of sulphuric acid which reaches the vinegar in the above manner is exceedingly small, and, moreover, is partially fixed to the mineral bases (lime and magnesia) contained in the water used in the preparation of the alcoholic liquid. Hence a manufacturer who sulphurs his barrels need not fear being accused of having adulterated his vinegar by the direct addition of sulphuric acid. Sulphured vinegar must be stored at least several weeks before it is salable, the odor of sulphurous acid adhering to it perceptibly, and disappearing only at the rate at which the sulphurous acid is converted into sulphuric acid.

*Fining Vinegar.*—Similar to wine, vinegar can be obtained bright by “fining” with isinglass. This method is employed by a number of manufacturers though it offers no advantages as compared with filtration. The isinglass to be used is prepared as follows: Cut with a pair of scissors into narrow strips  $\frac{1}{2}$  to 1 drachm of isinglass for every 20 gallons, and soak it in water in a porcelain dish for 24 hours. The resulting jelly-like mass is pressed through a linen cloth. A solution of  $\frac{1}{3}$  to  $\frac{2}{3}$  drachms of tannin for every 20 gallons is then added to the isinglass and the mass diluted with vinegar. The whole is then thrown into the barrel and thoroughly mixed with the contents. The clarified vinegar is finally drawn off from the sediment.

*Coloring Vinegar.*—Vinegar prepared from alcohol is limpid as water or only slightly colored. Prior to the general introduction of the quick process consumers were accustomed to the dark yellow product prepared from wine or beer, and many are still prejudiced against slightly colored vinegar, considering it weaker. Unfounded as this prejudice is, the manufacturer is nevertheless obliged to recognize it, and to suit the public taste, must color his vinegar by artificial means. Caramel or burnt sugar prepared from glucose is a simple and perfectly harmless coloring. It is made by melting the glucose in a shallow iron vessel over a fire, stirring constantly with a long-handled spoon. The melted mass soon turns



brown and rises in the vessel. The conversion into caramel being hastened in the presence of alkalies, the addition of a small quantity of pulverized carbonate of ammonium—about  $1\frac{1}{2}$  to 2 per cent. of the weight of the glucose used—is of service at this stage. The mass is now heated with constant stirring until it becomes black, runs from the spoon in viscous dark brown threads, and a sample dropped upon a cold surface congeals to a black mass impervious to light except upon the edges. The vessel is then lifted from the fire and the contents poured out upon metal or stone plates. The taste of the congealed mass should not be bitter, or at least only slightly so. On exposure to the air, the caramel deliquesces to a thick black fluid, and, therefore, it should immediately after its preparation, be converted with water into a solution of the consistency of syrup, such concentrated solution keeping better than a dilute one which readily molds. Immediately before use the solution is diluted with water, and enough of it added to the vinegar to give it the desired coloration. Some manufacturers use molasses or dark syrup for coloring vinegar.

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## CHAPTER XVI.

### PREPARATION OF VINEGAR FROM VARIOUS MATERIALS.

SINCE acetic acid is formed by the oxidation of alcohol, vinegar can, of course, be prepared from every kind of fluid containing alcohol, such as beer, wine, cider, as well as from the juice of sacchariferous fruits which has passed into alcoholic fermentation. By allowing grain to germinate, a body to which the term *diastase* is applied is formed, which possesses the property of converting starch into fermentable sugar and dextrin when brought in contact with it under certain conditions. Vinegar can, therefore, be prepared from starch—though in a round-about way—by treating the latter with

grain containing diastase (malt), whereby it is converted into maltose and dextrin. The fluid (sweet mash) is compounded with yeast, and the sugar—and with a correct execution of the process the dextrin also—is converted into alcohol by vinous fermentation. The resulting alcoholic liquid can then be used for making vinegar.

Alcohol or spirits of wine obtained in the above-described manner from the starch contained in potatoes or grain being at present the chief material used in the manufacture of vinegar, the greater portion of the latter brought into commerce might actually be designated potato or malt vinegar according to the elementary material used. The great progress made in modern times in the preparation of malt, brewing of beer, and in the distilling industry has been accompanied by a constantly extending division of labor. While formerly every brewer and distiller prepared his own malt, there are at present numerous establishments exclusively engaged in this branch of the industry which sell their product to the brewer and distiller. The manufacturer of vinegar who did not use materials containing finished alcohol (beer or wine) had to undertake the laborious work of making the malt and preparing and fermenting the mash in order to obtain an alcoholic liquid which he could finally convert into vinegar. With the present improvements in the preparation of malt and the production of alcohol, the vinegar manufacturer can work more cheaply by buying the alcohol, and the manufacture of so-called malt or grain vinegar would pay only where heavy taxes prevent the direct use of alcohol.

Formerly, when, in consequence of defective processes, many a brewing or batch of malt spoiled, it was used for making vinegar. But, as a rule, the vinegar obtained was not of a fine taste and remained turbid, and besides, the operation was frequently interrupted by all sorts of incidents, which led to the opinion of malt-vinegar not possessing keeping properties.

Beer-wort judged by its composition does not seem a suit-

able material for making vinegar. Besides a certain quantity of fermentable sugar (maltose), it contains a considerable amount of dextrin and other fermentable bodies. For the purpose of making vinegar the maltose alone can be considered, it being the only fermentable constituent of beer-wort. Hence, vinegar prepared from beer-wort always contains a considerable quantity of dextrin and extractive substances, and, consequently, is of a more thickly fluid nature than belongs to vinegar, and clarifies with difficulty. Moreover, this drawback exerts a disturbing influence upon the behavior of the vinegar when stored, it being frequently changed by further processes of fermentation into a slimy fluid, and acquires an insipid taste and loses a large portion of its content of acetic acid.

Alcoholic mashes containing in consequence of faulty preparation a considerable quantity of dextrin show, when used for making vinegar, a behavior similar to that of beerwort; the vinegar obtained clarifies with difficulty and does not keep well. Fermented whiskey-mashes properly prepared contain, however, only very small quantities of dextrin and extractive substances, and, when freed by filtration from admixed husks, can be used as a material for the manufacture of vinegar and yield an entirely normal product.

According to experience, the process of the formation of vinegar proceeds in the most uniform manner by preparing the alcoholic liquid from dilute alcohol, and, consequently, in a vinegar factory connected with a distillery it would be best to dilute *non-rectified* spirits of wine with the required quantity of water and add from 10 to 20 per cent. of the weight of the alcoholic liquid of fermented mash. The latter containing salts and nitrogenous substances suitable for the nourishment of the vinegar ferment serves, in this case, as a substitute for the beer generally used in vinegar factories for the preparation of alcoholic liquid.

*Manufacture of Malt or Grain Vinegar.*—Under certain local conditions the manufacture of vinegar from malt, with or with-



out an addition of grain, can be profitably carried on in connection with that of compressed yeast. Such factories for evident reasons not being established on an extensive scale, a description of the preparation of vinegar in connection with that of compressed yeast without the use of expensive machinery will be given.

The preparation of the fundamental material, malt, requiring much labor and knowledge, it will be best for the manufacturer to buy the article already prepared. Malt kiln dried at as low a temperature as possible and yielding a light-colored extract when treated with warm water should be chosen. Many malt houses prepare such malt especially for distilling purposes. Malt prepared for brewing purposes is after the actual kiln-drying heated to a temperature frequently exceeding 158° F. for the formation of certain aromatic combinations and coloring substances which are to impart to the beer a specific taste and dark color. Independently of the dark color of the vinegar prepared from such malt, it contains a considerable quantity of dextrin, and consequently acquires an insipid by-taste, clarifies with difficulty, and is readily subject to injurious alterations. Malt, as is well known, contains diastase, which in mashing with water effects the conversion of the starch into maltose and dextrin. By kiln-drying at a very high temperature a portion of the diastase is, however, rendered ineffective, and in mashing comparatively little maltose but a large quantity of dextrin is formed. Mashing, in this case, would have to be continued for a long time in order to obtain a larger quantity of maltose.

With the use of but slightly kiln-dried malt, in which the efficacy of the diastase has not been injured by a high temperature, the greatest directly obtainable quantity of maltose and the smallest amount of dextrin are procured. The proportion of maltose to dextrin is in this case as 4 : 1, or in other words, the finished mash contains about 80 per cent. of maltose and 20 per cent. of dextrin. The dextrin cannot be directly converted into acetic acid by the vinegar ferment and

consequently would be found in the finished product. It is, however, possible to treat the finished mash in such a manner that the total quantity of dextrin contained in it can be converted into maltose and the latter into alcohol. In this case the theoretically calculated yield of vinegar from the malt will be nearly approached in practice, and the product thus obtained contain only a small quantity of extractive substances of the malt which are not decomposed by alcoholic or acetic fermentation.

Before entering upon a description of the mashing process, the theoretical part in mashing will be briefly discussed. Malt contains starch and diastase. By bringing the comminuted malt in contact with water of about  $131^{\circ}$  to  $133^{\circ}$  F., the starch is formed into paste and the diastase passes into solution. By the action of the diastase upon the starch, the latter is converted into maltose and dextrin, the finished mass containing 80.9 per cent. of maltose and 19.1 of dextrin. For reasons given later on, the finished mass is heated for a short time to between  $140^{\circ}$  and  $141.8^{\circ}$  F., without, however, exceeding this temperature, and then cooled off to the degree required for the induction of alcoholic fermentation.

Mash prepared in this manner contains, besides the stated quantities of maltose and dextrin, *effective* diastase, *i. e.*, such as possesses the power of liquefying starch. By heating to above  $158^{\circ}$  F. the diastase entirely loses this property. By compounding a mash of this nature with yeast, the diastase with the simultaneous action of the yeast is able to convert all the dextrin present in the fluid into maltose, and consequently the total quantity of starch originally present is converted into alcohol by this peculiar process, to which the term *after-effect of the diastase* has been applied.

Unmalted grain being cheaper than malt and the latter containing sufficient diastase to convert a very large quantity of starch into maltose and dextrin, a mixture of malt and unmalted grain (equal parts of both;  $\frac{2}{3}$  grain and  $\frac{1}{3}$  malt, etc.) can be used instead of malt alone. The latter is, however,

preferable for the manufacture of vinegar, it yielding a product of a finer taste than unmalted grain. The mode of preparing the mash is exactly the same as for the distillation of alcohol, and as the necessary information can be obtained from any treatise on that subject, only a brief sketch of the operation will here be given.

The malt carefully ground is mixed with cold water to a thin paste, which is stirred until all small lumps are dissolved. This mixing of the ground malt with water, *doughing in* as it is called, can be effected with the assistance of a crutch or rake, but best in a vat provided with a mechanical stirring apparatus.

Doughing-in being finished, water of  $140^{\circ}$  to  $149^{\circ}$  F. is permitted to run in until the mash shows a temperature of about  $131^{\circ}$  to  $133^{\circ}$  F. During this operation the mash should be constantly stirred. The at first thickly fluid mass will soon be observed to become thinly fluid by the starch paste being converted into soluble bodies. Mashing is finished in 2 to  $2\frac{1}{2}$  hours, and will be the more complete the more accurately the temperature is maintained at  $131^{\circ}$  to  $133^{\circ}$  F. The completion of the process is recognized by a filtered sample cooled to the ordinary temperature remaining colorless after the addition of iodine solution.

The mash having reached this state, sufficient hot water is added with constant stirring to raise the temperature to  $140^{\circ}$  or  $141.8^{\circ}$  F. The purpose of this operation is to render all ferments present in the mash ineffective. Lactic acid ferment and frequently also butyric acid ferment always adhere to the malt, and, if allowed to develop in the mash, would form lactic and butyric acids during fermentation which would be injurious to the process of alcoholic fermentation as well as to the properties of the vinegar to be manufactured. The mash is now reduced to a temperature of about  $57^{\circ}$  or  $59^{\circ}$  F. by bringing it into the cooling-back or passing it through a system of refrigerating pipes. When working on a small scale the mash can be suitably cooled by allowing cold water to pass through a coil placed in a vat containing it.



The strength of the vinegar to be manufactured depends on the concentration of the mash; mashes showing a saccharometer statement of 20 per cent. contain after fermentation about  $9\frac{1}{2}$  per cent. of alcohol which yields vinegar of about 8 per cent.; mashes showing 18 per cent. yield vinegar of about 7 per cent., so that 1 per cent. of acetic acid in the vinegar may be calculated on for about every  $2\frac{1}{2}$  degrees indicated by the saccharometer.

The mash is now set with yeast, though the latter may be added when the mash still shows a temperature of  $71.5^{\circ}$  to  $75^{\circ}$  F., the yeast having then time to vigorously propagate. Mashes prepared from malt alone are uncommonly rich in nourishing substances for the yeast, the latter propagating abundantly and inducing a very vigorous process of fermentation. This can be profitably utilized by combining the manufacture of vinegar and that of compressed yeast, a valuable product being thus obtained without any extra expense and with but little labor. At a certain stage of the alcoholic fermentation the yeast comes to the surface of the fluid and can be lifted off. By washing the yeast once or twice with cold water and then freeing it from the excess of water by pressing, compressed yeast is obtained which, with the exception of the portion to be used for setting fresh mashes, can be sold.

Up to the completion of alcoholic fermentation the treatment of the mash as can be seen from the preceding description, does not essentially differ from that to which mashes for the manufacture of alcohol are subjected. If, however, the completely fermented "ripe" mash is to be used for making vinegar, it should be subjected to a special treatment, the object of which is to prepare a fluid containing no living yeast.

By filtering the mash through a closely woven linen cloth the particles of malt-husks, etc., are retained but not the cells of alcoholic ferment which may be present, and which, on account of their minuteness, are difficult to separate from the fluid by filtration. It is, therefore, best to heat the mash before filtration to about  $140^{\circ}$  F. whereby the ferment is killed,

and at the same time a certain quantity of albuminous substances dissolved in the fluid is rendered insoluble and separated. The heating of the mash is best effected by passing it through a coil of tin-pipe placed in a boiler filled with water kept constantly boiling. The temperature of the fluid can be readily regulated by increasing or decreasing the velocity with which it passes through the coil. If the fluid heated to  $140^{\circ}$  F. were allowed to cool in the air, a large portion of the alcohol contained in it would be lost by evaporation, and it is therefore allowed, after heating, to pass through a second coil of pipe which is surrounded by cold water whereby it is cooled to at least  $86^{\circ}$  F. This fluid is then filtered through a linen bag, is being repeatedly poured back into the filter until it runs off sufficiently clear. It will not, however, be obtained perfectly clear in this manner, the yeast cells being too minute to be retained by such a filter, but having been killed by heating, their presence in the fluid is connected with no disadvantage.

By mixing the filtered fluid with from 10 to 15 per cent. of its volume of vinegar, an alcoholic liquid is obtained which can be worked in the usual manner in the quick-process generators, and yields an agreeable aromatic vinegar which clarifies rapidly and improves by storing.

According to the slow process, the fermented malt-wort is run into casks placed in apartments called "stoves," since they are heated by stoves or steam to a temperature ranging from  $70^{\circ}$  to  $80^{\circ}$  F. The casks are arranged in parallel rows, resting upon long wooden beams elevated about 18 inches from the ground, and having their bungs uppermost while a small hole on top of the front head of each causes the circulation of air.

A large saving of labor will be effected by connecting elevated tanks holding the fermented wort with pipes and movable flexible hose which will allow of the rapid and easy filling of the casks. The vinegar produced is siphoned off into inclined troughs, which deliver it to a central underground tank, from which it is pumped into the storing tanks.

Malt vinegar generally contains a great deal of mucilaginous matter which settles with difficulty, preventing its keeping, while giving nourishment to vinegar eels. It is therefore necessary to filter it, and for this purpose it is pumped into the *refining* or *rape* vessels. These vessels are often filled with wood shavings, straw, or spent tanner's wood, but nothing acts as well in producing by filtration a clear bright vinegar as the stalks and skins of grapes or raisins technically called "*rape*." Where there is power and a large quantity of vinegar is manufactured, the filtering is effected under a considerable hydrostatic pressure. The *rape* is placed in a closed vessel between two false perforated bottoms. A circuit of pipes is connected at the lower and upper part of the vessel, and by means of a pump the vinegar is made to pass again and again through the *rape*.

The mode of manufacture is frequently effected by "*fielding*." In this case, as the term implies, the process is conducted in the open air. The casks rest on small frames  $1\frac{1}{2}$  feet high, being supported by firm pillars of brick-work or wood. The operation generally begins in spring and continues during the summer. The fermented liquor is run into the casks by the bung-holes, the latter being left open in dry, and loosely covered with a tile in wet weather. Gradually the alcohol of the "*gyle*," as the fermented liquor is called, becomes oxidized, and acetic acid is produced, of course simultaneously affording vinegar. The latter is then drawn off and transferred to the refining or *rape* vessels where it passes through the process of filtration already described.

In some factories large quantities of sour ale and beer are converted by similar processes into vinegar, but the product is much inferior to the vinegar made from malt-wort. The large amount of nitrogenous and other extractive substances which those liquids contain undergoes a second or putrid fermentation after the alcohol has been oxidized into acetic acid, and in doing so reacts upon the acid, leaving a liquid of a disagreeable odor slightly resembling very stale beer. By the



addition of sulphuric acid this second fermentation is postponed for some time, but the vinegar has nevertheless a nauseous smell which renders it objectionable.

*Vinegar from Sugar-Beets.*—The juice of the sugar-beet contains a considerable quantity of cane-sugar and is readily brought into alcoholic fermentation, so that seemingly this root would form a very suitable material for the manufacture of vinegar. Sugar-beets contain on an average 12 per cent. of cane-sugar, the latter yielding, when completely fermented, a fluid containing about  $6\frac{1}{2}$  per cent. by weight of alcohol; a fluid with this percentage of alcohol yields vinegar with 6 per cent of acetic acid.

In addition to sugar the juice of the beet-root contains, however, a large number of other substances which exert an influence upon the course of alcoholic fermentation, and, besides alcohol, a large quantity of fusel oils is formed, so that the alcohol has to be thoroughly rectified before it is fit for use. The fermented beet-root juice itself has, however, a disagreeable taste and odor, and the vinegar prepared from it showing similar properties will not be fit for household purposes until a remedy for these drawbacks is found. Numerous experiments made for the purpose of freeing beet-root vinegar from the substances which impart to it a disagreeable odor and taste have given no favorable results. Filtering through charcoal, and even distilling the vinegar and treating the distilled product with strongly oxidizing bodies, do not produce the desired effect. From these experiments it would seem impossible to directly obtain from sugar-beets vinegar fit for household use.

*Vinegar from Sugar, Fruits and Berries.*—By fermenting sugar solution with pure yeast and pouring off the clear alcoholic fluid, the latter shows a slightly acid reaction (from succinic acid), but is not converted into vinegar even if standing for several weeks in the most suitable temperature, because the vinegar ferment is wanting. By adding, however, an excess of yeast, so that it remains partially suspended in the

fluid, which can be effected by the addition of solution of gum or starch paste, the nourishment for the spores of the vinegar ferment reaching the fluid from the air is provided and acetification takes place.

Cadet-Gassicourt advises the fermentation together of 124 parts of sugar, 868 of water, and 80 of yeast, and to filter after one month. Or, according to another formula: Sugar 245 parts, gum 61, water 2145, yeast 20. Allow to ferment at 68° F. Fermentation begins the same day and is completed in 15 days.

Doebereiner gives the following directions: Dissolve 10 lbs. of sugar in 180 quarts of hot water, add 6 lbs. of pulverized crude tartar (it dissolves only partially), and after cooling to 77° F. induce fermentation by  $4\frac{1}{2}$  quarts of beer yeast. In about eight days, when fermentation is finished, add about 15 quarts of spirits of wine of at least 50 per cent. Tr. or 8 quarts of alcohol of 90 per cent. Tr., and bring the mixture into the acetifying vessel. This fluid would also be suitable for the quick process.

For making vinegar on a small scale for domestic use, brown sugar with water alone, or sugar with raisins, currants, and especially ripe gooseberries, may be used. These should be mixed in the proportion which would give a strong wine, put into a small barrel filled to about three-fourths of its capacity, and bunged very loosely. Some yeast should be put in and the barrel set in the sun in summer or a little way from the fire in winter, and fermentation will soon begin. This should be kept up constantly, but moderately, till the taste and smell indicate that the vinegar is complete. It should then be poured off clear, and bottled carefully. It will keep much better, if it is boiled for a minute, cooled, and strained before bottling.

With the exception of apples and pears, the different varieties of fruit cannot be had in such abundance as that they could be used for the manufacture of vinegar on a large scale, and hence only a brief description of their utilization for that purpose will be given.

It is characteristic of most of our varieties of fruits, and especially of berries, that in proportion to their content of sugar they have a much greater content of free acids than grapes, and this circumstance must be taken into consideration, as otherwise wine would be obtained which contains a considerable quantity of unfermented sugar. The following table shows the average content of sugar and free acid in the most common varieties of fruits:—

	Sugar.	Free acid calculated as malic acid.
Cherries . . . . .	10.00	—
Apples . . . . .	6.25 to 13.99	0.691
Pears . . . . .	8.78	—
Currants . . . . .	6.40	2.147
Strawberries . . . . .	5.09 to 11.31	1.363
Gooseberries . . . . .	6.93	1.603
Bilberries . . . . .	5.78	1.341
Raspberries . . . . .	4.02	1.484
Blackberries . . . . .	4.44	1.188

According to the above table, currants, gooseberries, raspberries, etc., contain on an average scarcely 6 per cent. of sugar, and consequently their juice, after complete fermentation, would give a fluid with about 3 per cent. of alcohol, from which vinegar with about  $2\frac{1}{2}$  per cent. of acetic acid could be obtained. Such vinegar being, however, too weak, those berries would not seem suitable for the direct preparation of vinegar. Moreover, the complete fermentation of the juice of most berries is very difficult, the free acids, among which malic acid preponderates, exerting an injurious influence upon the progress of fermentation.

Vinous fluids of an agreeable taste can, however, be prepared from berries, and from them an aromatic and finely flavored vinegar, by decreasing the content of acid in the juice and increasing that of sugar. The juice of currants, as seen from the above table, contains in round numbers 6 per cent. of sugar and 2 per cent. of malic acid. By diluting this juice with an equal volume of water a fluid containing 3 per cent.



of sugar and 1 per cent. of acid is obtained, and the content of the former can be increased at will by the direct addition of sugar.

By compounding, for instance, 100 quarts of currant juice with 100 quarts of water and adding 34 lbs. of sugar, the resulting fluid contains about 20 per cent. of sugar and after complete fermentation gives a fluid with about 9.5 per cent. of alcohol, which yields vinegar of nearly 9 per cent. strength. The taste of this vinegar is, however, stronger and more agreeably acid than that of vinegar from alcohol, it containing besides acetic acid about 1 per cent. of malic acid. Moreover, vinegar obtained from berries contains a certain quantity of extractive substances and odoriferous products of fermentation, so that it possesses an agreeable bouquet and thus appears more valuable than the ordinary product.

In many regions bilberries grow in abundance and can be bought very cheap. Treated in the above manner, they yield an excellent vinegar, possessing, however, a somewhat harsh by-taste, due to the tannin contained in the berries. The latter can be removed from the fermented fluid before using it for the preparation of vinegar, by compounding the latter when quite clear with gelatine solution or fresh white of egg, both forming insoluble combinations with the tannin, which separates in the form of flakes.

In regard to the preparation of vinegar from berries, it remains to be remarked that, after pressing the bruised berries, the juice is compounded with water and sugar and at once brought into fermentation by the addition of yeast (best fresh wine-yeast, or if this be wanting, compressed yeast divided in water). Fermentation should take place at quite a high temperature, 68° to 72° F. The separated yeast is carefully removed from the fermented liquid and the latter stored away in barrels kept constantly filled up to the bung, or at once used for the preparation of vinegar. By converting fruit-wine into vinegar by means of the vinegar ferment floating upon the fluid a much finer product is obtained than by the quick process.

*Peaches as Vinegar Stock.* Mr. H. C. Gore\* has made experiments regarding the value of peaches as vinegar stock. The conclusions drawn by him from this work are, first, that peaches contain sufficient fermentable sugar for use as vinegar stock, and, second, that they can be successfully handled by machinery already in use for making apple cider and vinegar. Other points of interest are as follows: First, but little variation was found in the composition of the same variety of peaches when obtained from different localities. Second, the peach juices analyzed were found to be richer in sugar than those which have been previously analyzed by others, but they were about 1 per cent lower in sugar than average apple juices. They were considerably richer than apples in sucrose and in acid. Third, it was found that the use of pure culture yeasts was not necessary to insure rapid alcoholic fermentation. Fourth, the ciders prepared from peaches were considerably poorer in alcohol than apple ciders on account of the fact that peaches contain less total sugars than apples. Fifth, the presence of brown rot was found not to interfere with the alcoholic fermentation of the ground peaches, but a large proportion of the sugars was wasted by allowing the fruit to rot before fermenting. Sixth, well-flavored vinegars were produced by the use of a small quick-process generator. These vinegars were of acceptable quality, though turbid, and did not possess the distinctive peach flavor.

*Cider Vinegar.* The manufacture of cider itself will be described in another portion of this work and, hence, its utilization for the preparation of vinegar will here only be given.

The preparation of vinegar from good cider is not difficult, the process of acetification by means of the vinegar ferment floating upon the surface yielding an aromatic product of a fine flavor which is nearly of as good a quality as wine vinegar. On account of its content of malic acid, the vinegar is

\* United States Department of Agriculture, Bureau of Chemistry—Circular No. 51.

more acid than ordinary vinegar with the same content of acetic acid. But in order to produce cider vinegar of the first quality one must have good cider; vinegar made of watered cider will be thin and weak.

The cider extracted by the first pressing of the apples is but in rare cases used for making vinegar, the juice obtained by subjecting the pomace, with the addition of water or sugar solution, to a second and third pressure being as a rule utilized for the purpose. The juice thus obtained should be so constituted as to yield vinegar containing  $4\frac{1}{2}$  to  $5\frac{1}{2}$  per cent. of acetic acid. The cider to be converted into vinegar should be as clear as possible and, if necessary, filtration over sand or storing for some time is advisable.

The conversion of cider into vinegar is best effected in a generator furnished with a tilting trough for the intermittent supply of cider.

After the cider has been extracted and the cheese removed from the press, the pomace may also be utilized for making vinegar by treating it as follows: The pomace is piled up on a platform of suitable construction and allowed to ferment. In the course of a few days considerable heat will be developed, when a few pailfuls of warm water (not boiling) are poured upon the pile, and in the course of twenty-four hours the pomace will be in proper condition for grinding. It is then run through a grater-mill and relaid upon the press in a cheese in the same manner as originally laid in cider making. It is then subjected to heavy pressure until the liquid contained in the cheese is extracted. This liquid may be exposed in shallow open casks in a warm room, and in a short time will be found good vinegar; or it may be immediately passed through a generator.

Mr. Walter G. Sackett \* gives directions for *home-made cider vinegar* as follows: "The sweet cider as it comes from the

\* Bulletin 192, November, 1913. The Agricultural Experiment Station of the Colorado Agricultural College.



press may either be placed at once in barrels, which should not be filled more than two-thirds or three-fourths full, or if one has suitable wooden tubs or vats, in a clean, cool place; it may be stored there from 12 to 24 hours to permit settling, after which it should be transferred to barrels. The bung should be left out and a loose stopper of cotton batting inserted in the hole to decrease evaporation and prevent dirt from falling in. The barrels should not be tightly stoppered until the vinegar contains at least 4 to 5 per cent. of acetic acid, at which time they should be filled entirely full and securely bunged. Throughout the entire period of vinegar making, the casks should be placed on their side and not on the end. This gives the cider a larger free surface exposed to the air, which is quite essential to rapid vinegar formation. It may also be of some advantage in admitting air to bore a  $1\frac{1}{2}$  inch hole in each end of the barrel along the upper edge. If this is done, the holes should be covered with fine gauze wire or two thicknesses of cheese-cloth to exclude small vinegar flies.

“A few days after the cider is put into the barrels the characteristic frothing appears at the bung-hole. To use a common expression, ‘it is beginning to work.’ This indicates that the alcoholic fermentation, the first step in the vinegar-making process, has begun, and the sugar of the apple juice is being converted into alcohol and carbon dioxide gas. To depend upon the wild yeast of the air to accomplish the fermentation is too uncertain since many of them are able to convert only a small part of the sugar into alcohol, while others act so slowly that they are impracticable. Inasmuch as the percentage of acetic acid in the vinegar depends directly upon the amount of alcohol produced, it is very essential to secure as large a yield of alcohol as possible from the sugar present. This means converting all of the sugar into alcohol in the shortest time possible. The most satisfactory way of doing this is to add one cake of compressed yeast, stirred up in a little cooled, boiled water, to each five gallons of sweet cider.

In place of this, one quart of liquid wine yeast, propagated from a pure culture, may be used for each thirty gallons of cider.

“During the alcoholic fermentation, the cider should be kept at a temperature of 65° to 80° F. Here is where many make the very serious mistake of putting their fresh cider into a cool cellar where the fermentation takes place entirely too slowly. If the cider is made in the fall, the barrels should be left out of doors for a while on the protected, sunny side of a building and kept warm, unless a regular vinegar-cellar, artificially heated, is at hand.

“If yeast is added and the proper temperature is maintained, the alcoholic fermentation should be completed in six weeks to three months in place of seven to ten months as in the old-fashioned way. Experiments along this line have shown that when yeast is added and a temperature of 70° F. is held, the cider at the end of one month contained 7.25 per cent. of alcohol as against .11 per cent. when no yeast was used and the temperature was between 45° and 55° F. Cider kept in a cellar at 45° to 55° F. with no yeast added required seven months to make 6.79 per cent. of alcohol.

“Temperature, alone, is an important factor as shown by an experiment wherein cider to which no yeast was added was held for three months at 70° F. and yielded 6.41 per cent. of alcohol.

“There is no question but that the time required for completing the alcoholic fermentation can be reduced at least one-half by adding yeast and by maintaining the proper temperatures. By hastening this operation, the loss of alcohol by evaporation is reduced, and the acetic fermentation can be started that much sooner.

“As soon as alcoholic fermentation is completed draw off the clear liquid, being very careful not to disturb the sediment in the barrel. Wash out the barrel thoroughly and replace the hard cider. It is believed that removing this sediment permits the acetic acid to form somewhat more quickly,

and furthermore, the sediment may undergo decomposition and impart a disagreeable flavor to the cider. Again these dregs may harbor living bacteria which either destroy acetic acid or interfere with its formation.

"We are now ready to introduce the acetic acid germs. This may be carried on in a number of different ways, but preferably by means of a pure culture of a desirable organism which has been selected because of its ability to produce strong acetic acid and impart an agreeable flavor to the vinegar. In place of the pure culture starter, one may add two to four quarts of good cider vinegar containing more or less 'mother' for each barrel. The introduction of a desirable organism is left to chance in this case. A serious objection to the latter method is that sometimes one introduces foreign organisms with the 'mother' which may prove detrimental to the vinegar. Pure culture\* is free from this objection.

"With the acetic fermentation, as with the alcoholic, the higher temperatures favor the changes. Experimental work shows that hard cider to which no acetic acid bacteria were added other than those that came from the air, and kept at 65° F., when six months old, contained 7.03 per cent. of acetic acid, while that held at 55° F. showed only 3.63 per cent.

"The addition of some kind of an acetic acid starter, either as a pure culture of the acetic organism or as good vinegar, hastens the fermentation and reduces appreciably the time required for making marketable vinegar.

"For most satisfactory results we would recommend using the pure cultures and holding the vinegar at a temperature of 65 to 75° F. Under these conditions, salable vinegar can be obtained in three to six months in place of two to three years, as is often the case. Theoretically, 100 parts of alcohol should give about 130 parts of acetic acid, but in actual practice this will probably fall below 120.

\* The pure cultures, both of yeast and acetic acid bacteria, for vinegar making, here referred to, can be obtained by addressing The Bacteriological Department, Experimental Station, Fort Collins, Colorado.



“When the acetic acid has reached 4.5 to 5 per cent., fill the barrels as full as possible and cork tightly. In this way, contact of the air with the vinegar is cut off and the acetic acid organisms soon cease their activity. If this is not done and the acetic and other bacteria are allowed to develop indefinitely, there is apt to be a reverse reaction resulting in a partial or complete loss of the acetic acid. Such vinegar is, of course, worthless.”

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## CHAPTER XVII.

### VINEGAR SPECIALTIES.

THESE specialties may be divided into two groups: Into those with a specific odor, and those with a specific odor and taste. As an example for both kinds tarragon vinegar may be taken. If it is prepared by simply dissolving in the vinegar the volatile oil of dragon's wort (*Artemisia dracunculus*) obtained by distillation with water, the product is simply *perfumed* vinegar, the odor of the volatile oil being mixed with that of the acetic acid, but the taste remains unchanged. If, however, the fresh leaves of the plant are macerated with vinegar, not only the volatile oil is dissolved, but also certain extractive substances peculiar to this plant, and the taste of the vinegar is also changed, the product in this case being *aromatized* vinegar.

By dissolving in vinegar rose oil or rose water (perfumed), rose vinegar is obtained. By treating raspberries with vinegar the latter absorbs not only the odoriferous substances of the raspberry, but also the non-odoriferous extractive substances, and the product is aromatized vinegar.

By skillful manipulation every volatile oil can be dissolved in vinegar, and consequently as many different varieties of perfumed vinegar can be prepared as there are volatile oils.

In fact, perfumers prepare a number of such varieties which contain one or more volatile oils whose odors harmonize and are sold as volatile spirit of vinegar, fumigating vinegar, etc. Such vinegars can be prepared in various ways, the finest odors being, however, obtained by distilling the fresh parts of the plants with water and mixing the distillate, which actually represents a solution of the volatile oil in water, with strong vinegar. The finest rose vinegar, orange blossom vinegar, etc., are prepared in this manner.

For this rather tedious process of preparing perfumed vinegar, the one in which *freshly* prepared volatile oils are used may be advantageously substituted. To be sure the volatile oils dissolve only sparingly in vinegar, but sufficiently so to impart their characteristic odor to it. By using an excess of volatile oil it does not dissolve, but distributes itself in fine drops throughout the vinegar, rendering the latter opalescent, so that fining with tannin and isinglass is necessary to make it bright again.

This drawback can be avoided by a simple manipulation which is based upon the fact that a body dissolving with difficulty dissolves the more readily the greater surface it offers to the solvent.

Prepare glass-powder as fine as the best wheat flour by heating pieces of glass, throwing them into cold water, and pulverizing and elutriating in a mortar. By the sudden cooling the glass becomes so brittle that it can be readily converted into a fine powder. Bring a suitable quantity of this powder into a porcelain dish and drop volatile oil upon it with constant rubbing until it is uniformly moistened. Pour the vinegar to be perfumed upon this glass powder and stir gently with the pestle. The fluid is then poured into the barrel intended for the reception of the perfumed vinegar and a fresh quantity of vinegar poured upon the glass-powder, this being continued until all the glass-powder has been brought into the barrel by stirring and pouring over fresh vinegar. The barrel is then entirely filled with vinegar, and after being securely bunged,

rolled in order to secure a uniform mixture of its contents. It is then allowed to rest for a few days for the glass-powder to settle. The entirely clear perfumed vinegar is then drawn off into bottles, which are to be kept in a dark cool room, the odor of the volatile oil being injured by light and heat.

For the preparation of volatile fumigating or toilet vinegars it is best to dissolve the volatile oils in uncolored vinegar prepared from alcoholic liquid. Where the remaining of a small residue after the volatilization of the perfumed vinegar is of no importance, pulverized sugar may be substituted for the glass-powder, as it acts in the same manner; the only difference is that the glass-powder being an insoluble body falls to the bottom of the barrel, while the sugar dissolves together with the volatile oil in the vinegar.

By the above-described process perfumed vinegar with the odor of dragon's-wort, peppermint, anise, rose, etc., etc., may be prepared, and by a suitable mixture of those whose odors harmonize, a great number of fumigating and toilet vinegars may be obtained.

The preparation of aromatized vinegars by means of the extractive substances of plants is very simple. The parts of plants to be extracted are placed in a suitable vessel, a barrel or large flask, and after pouring vinegar over them and closing the vessel, are allowed to rest for a few weeks in a moderately warm room. In case glass vessels are used they have to be kept in a dark room, light exerting an injurious influence upon the odors. The vegetable substances used for aromatizing vinegar containing, as a rule, a large quantity of water, strong vinegar, with 10 to 11 per cent. acetic acid, should be used.

Below a few formulas for toilet and table vinegars are given :

#### TOILET VINEGARS.

*Mohr's Volatile Spirits of Vinegar.*—Equal parts of acetic acid and acetic ether, perfumed with a few drops of oil of cloves.

*Aromatic Vinegar.*—Concentrated acetic acid 8 ounces, oil of lavender 2 drachms, oils of rosemary and cloves each 1 drachm, oil of camphor 1 ounce.



Bruise the camphor and dissolve it in the acetic acid, then add the perfumes; after standing for a few days with occasional agitation it is strained and ready for use.

*Henry's Vinegar.*—Dried leaves of rosemary, rue, wormwood, sage, mint and lavender flowers each 1 ounce, bruised nutmeg, cloves, angelica root and camphor each  $\frac{1}{2}$  ounce, alcohol (rectified) 8 ounces, concentrated acetic acid 32 ounces.

Macerate the materials for a day in the alcohol; then add the acid and digest for a week longer at a temperature of about 59° F. Finally press out the now aromatized vinegar and filter it.

*Vinaigre des Quatre Voleurs.*—Fresh tops of common wormwood, Roman wormwood, rosemary, sage, mint and rue each  $\frac{3}{4}$  ounce, lavender flowers 1 ounce, garlic, calamus aromaticus, cinnamon, cloves, and nutmeg each 1 drachm, camphor  $\frac{1}{2}$  ounce, alcohol or brandy 1 ounce, strong vinegar 4 pints.

Digest all the materials, except the camphor and spirit, in a closely covered vessel, for a fortnight, at summer heat; then express and filter the vinegar produced and add the camphor previously dissolved in the brandy or alcohol.

*Hygienic or Preventive Vinegar.*—Brandy 1 pint, oils of cloves and lavender each 1 drachm, oil of marjoram  $\frac{1}{2}$  drachm, gum benzoin 1 ounce.

Macerate these together for a few hours, then add 2 pints of brown vinegar and strain or filter.

*Cosmetic Vinegar.*—Alcohol 1 quart, gum benzoin 3 ounces, concentrated aromatic vinegar 1 ounce, balsam of Peru 1 ounce, oil of neroli 1 drachm, oil of nutmeg  $\frac{1}{2}$  drachm.

#### TABLE VINEGARS.

*Anise Vinegar.*—Convert into a coarse powder anise seed 5 parts, caraway seed  $\frac{3}{5}$ , fennel and coriander seed each  $\frac{1}{5}$ , pour 5 parts of alcohol and 45 parts of strong vinegar over the powders, close the vessel air-tight and let the whole digest in a warm place for 6 to 8 days, shaking frequently. Then strain the liquid off, press out the residue, filter the vinegar, and put it up in bottles.

*Anchovy Vinegar.*—Reduce 1 pound of boned anchovies to a pulp in a mortar and pass the mass through a hair-sieve. The bones and parts which do not pass through the sieve are boiled for 15 minutes in a pint of water and strained. To the strained liquor add  $2\frac{1}{2}$  ounces of salt and the same quantity of flour together with the pulped anchovies, and allow the whole to simmer for 3 or 4 minutes; as soon as the mixture is cold add  $\frac{1}{4}$  pint of strong vinegar.

*Tarragon Vinegar.*—Pick the young tender leaves of dragon's-wort (*Artemisia dracunculus*) when the first flower-buds appear. Bruise the leaves, place them in a suitable vessel, pour good wine-vinegar over them, and let the whole stand for a few days. Then strain the vinegar through a cloth, filter and bottle. The bottles must be filled entirely full, as otherwise the vinegar will not keep.

*Compound Tarragon Vinegar.*—Comminute leaves of dragon's-wort 100 parts, common bean leaves 25, leaves of basil and marjoram each  $12\frac{1}{2}$ , bay leaves and orris root each 25, cloves  $3\frac{1}{3}$ , cinnamon  $6\frac{1}{2}$ , and shallots 25. Put all in a suitable vessel, pour 700 to 750 parts of pure, strong vinegar over it, let it stand in a warm place and digest 5 or 6 days, frequently agitating it. Then strain the vinegar through linen, press out the residue, add 25 parts of alcohol, and filter. Keep the vinegar in well-corked bottles in a cool, dark place.

*Effervescing Vinegar.*—Dissolve 500 parts of loaf sugar in 5000 parts of water, add lemon juice and rind cut up in the proportion of 1 lemon to 1 lb. of sugar,  $1\frac{1}{2}$  parts of the best cinnamon, and 12 parts of beer yeast thoroughly washed. Place the whole in a barrel, and after agitating it thoroughly let it ferment at a temperature of  $55^{\circ}$  to  $60^{\circ}$  F. When fermentation has ceased the vinous fluid is strained and mixed with 1000 parts of best wine-vinegar, previously boiled up, and yeast in the proportion of 1 spoonful to 5 lbs. of sugar. The fluid is then distributed in several earthenware pots and exposed to a temperature of  $77^{\circ}$  to  $88^{\circ}$  F. until it has been converted into strong vinegar. This, while remaining in the pots,

is mixed with 200 parts of French brandy and after two days bottled in small bottles. To each pound of this vinegar are added,  $\frac{2}{3}$  part of crystallized tartaric acid, pulverized, and  $\frac{1}{2}$  part of bicarbonate of soda. The bottles, as soon as the respective portion of the mixture has been added to each, must be corked as quickly as possible and then stored in a cool place.

*Herb Vinegar.*—Chop fine the leaves of marjoram and thyme each  $13\frac{1}{2}$  parts, common bean leaves  $6\frac{1}{2}$ , leaves of mint, basil and celery each  $3\frac{1}{2}$ , and fresh shallots  $1\frac{1}{2}$ . Pour 600 or 700 parts of good vinegar over the herbs and treat in the same manner as given for compound tarragon vinegar.

*Pine-apple vinegar.*—This excellent vinegar soon loses its flavor, and it is therefore best to prepare a small quantity at a time and keep in bottles closed air-tight.

Bruise the slices of pine-apple and pour over them a considerable quantity of vinegar. Close the vessel air-tight and let it stand 12 hours; then pour off the vinegar and filter.

*Celery Vinegar.*—Celery seed  $4\frac{1}{2}$  ozs., vinegar 1 pint. Digest 14 days; filter.

*Clove Vinegar.*—Cloves  $3\frac{1}{2}$  ozs., vinegar 1 pint. Digest 7 days and strain.

*Mustard Vinegar.*—Black mustard seed 2 ozs., vinegar 1 pint. Digest one week and filter.

*Lovage Vinegar.*—Lovage root 2 ozs., lovage seed 1 oz., vinegar 10 ozs. Digest one week and filter.

*Raspberry Vinegar.*—For the preparation of this vinegar it is best to use the residue remaining after pressing the ripe and crushed berries, as it contains sufficient aroma to impart to vinegar macerated with it for some time an agreeable odor and taste of raspberries. However, the juice may also be used, but if the vinegar itself is not very strong it becomes thereby too much diluted and consequently weak.

Crush the fresh berries to a paste and allow the latter to stand a few days, stirring it frequently, for the small quantity of sugar contained in the berries to ferment. By the alcohol



thus formed the pectin in the juice is to a great extent separated. The paste is then brought into a small bag and pressed.

The press-cake is crushed, made into paste with vinegar and spread out flat, exposed to the air for a few days, being frequently stirred. During this time the paste, at first pale red, again acquires, in consequence of a process of oxidation, a vivid red color. The quantity of vinegar required is then poured over the paste. The whole is then allowed to digest for a few days, when it is pressed and filtered. The flavor of raspberry vinegar is improved by adding 10 drops of acetic ether per quart. For 1 pound of pressed residue about 4 to 5 quarts of strong vinegar are used.

*Preparation of Acetic Ether.*—Among the numerous combinations into which acetic acid enters with other bodies, acetic ether is of special value for the vinegar manufacturer, it being directly used in the manufacture of vinegar. It is readily formed on alcohol coming in contact with acetic acid, and it would seem with special ease when the latter is in a nascent state. Hence a small quantity of it is found in nearly all red wines not prepared by fermentation in closed vats, its presence being due to the formation of a small quantity of acetic acid from the alcohol, which immediately combines with the ethyl oxide or ether.

In vinegar containing a small quantity of unchanged alcohol some acetic ether formed by the conversion of this alcohol into acetic acid is always present, and imparting a very delicate and agreeable bouquet to the vinegar, it is recommended to conduct the production of a fine article so that it contains a small quantity of it.

It is, however, not absolutely necessary to leave a small quantity of alcohol in the vinegar, as either acetic ether or alcohol can be directly added to the finished product. But in both cases the vinegar has to be stored for several weeks; in the first, for the purpose of harmonizing the odors of acetic ether and of acetic-acid, and in the latter, for the formation of acetic ether.

A fluid quite rich in acetic ether and very suitable for imparting bouquet to table vinegar can in a very simple manner be prepared by mixing in a flask one volume of highly concentrated acetic acid with 95 or 96 per cent. alcohol, and after closing the flask air-tight, allowing the fluid to stand in a warm room for several months. The resulting fluid is used as an addition to the vinegar whose odor is to be improved. Entirely pure acetic ether is best prepared in the following manner: To 9 parts of concentrated sulphuric acid 3.6 parts of commercial absolute alcohol are added by means of a funnel tube which reaches to the bottom of the vessel, at the same time keeping the liquid well stirred. After standing for 24 hours this mixture is added to 6 parts of sodium acetate which has previously been fused and broken in small fragments, and after 12 hours the mixture is distilled. Thus 6 parts of pure acetic ether are obtained, from which, by rectifying over calcium chloride, all traces of water are removed.

Pure acetic ether or ethyl acetate has the composition  $\left. \begin{array}{l} \text{C}_2\text{H}_5\text{O} \\ \text{C}_2\text{H}_3\text{O} \end{array} \right\}$

and represents a fluid clear as water with an agreeable but stupefying odor. Its specific gravity is 0.932 and it boils at 165.2° F. On account of its volatility it has to be kept in well-stoppered bottles, best in a cool place.

About 3½ to 7 ozs. of acetic ether suffice for the improvement of the odor of 100 quarts of vinegar.

## CHAPTER XVIII.

## MANUFACTURE OF WINE VINEGAR.

Since wine contains between 6 and 14 per cent. of alcohol, it evidently furnishes an excellent material for vinegar making. Both white and red wines may be used for the purpose, but as white wine vinegar is as a rule preferred, the product obtained from red wine is generally not salable until it has been decolorized, and the process of decolorizing impairs its flavor and aroma. Vinegar may also be made from grapes which are unsuitable for drying, shipping or wine making, and this may be the most profitable use, in some cases, to which even the best grapes can be put. If grapes are used they must of course be first made into wine by the usual process. According to Bioletti\* one ton of grapes of 20° Balling should on the average yield 135 gallons of vinegar of 9.8 per cent. acetic acid. It may be greater or less than this according as the grapes contain more or less sugar. This yield may be diminished by imperfect crushing and pressing of the grapes whereby more must is left in the pomace. Alcohol may be lost by imperfect or improper fermentation in which case the vinegar will be weaker. The greatest difference between the theoretical and the actual yield is in the change from wine into vinegar. This is because one or two per cent. of alcohol remains unconverted in the vinegar, and because during the process there is a considerable loss of alcohol and acetic acid by evaporation, and by reactions within the liquid which produce other substances at the expense of the alcohol and acetic acid. If the temperature during acetification is too high, or if the acetic bacteria are allowed to act too long, this loss may be much increased.

\* Grape Vinegar. By Frederic T. Bioletti. University of California Publications. Bulletin No. 227, 1912.



By allowing the crushed grapes to ferment on the skins before pressing, a somewhat larger volume of wine and therefore of vinegar may be obtained. This may amount to 150 or 160 gallons of vinegar from a ton of grapes. The vinegar, however, will be darker colored and, in the case of red grapes, red. This color can be removed, but the decoloration is difficult and involves some loss of quality.

Fermentation for twenty-four hours on the skins will much facilitate the extraction of the juice without, except in the case of grapes very rich in coloring matter, reddening the juice very much.

The question, what constitutes the superiority of wine vinegar over the ordinary product obtained from alcohol is not difficult to answer for those who have an accurate knowledge of the constitution of wine. Besides the ordinary (ethyl) alcohol, wine vinegar contains very small quantities of other alcohols, for instance, amyl alcohol, which in the same manner as ethyl alcohol is converted into acetic acid, are changed into acids possessing a peculiar odor. Moreover, wine very likely contains a series of odoriferous substances which produce its peculiar aroma termed bouquet or flower, the cœnanthic ether found in every wine forming, so to say, the keynote in the harmony of the odoriferous substances constituting the bouquet. In the conversion of wine into vinegar these bouquet substances are also changed in such a manner that bodies distinguished by a characteristic odor are formed. Furthermore, wine contains glycerin, a series of non-volatile organic acids, tartaric, malic, succinic acids, etc., and finally the so-called extractive substances. What changes these bodies undergo is not accurately known, but all of them are very likely subject to certain modifications because a smaller quantity of extractive substances and of non-volatile acids is found in the vinegar than in the original wine. The following table shows the composition of wine and of the vinegar formed from it:



Such wine cannot be used even for vinegar, the latter showing the same disagreeably bitter taste. Wine attacked by what is called "lactic acid degeneration" can be used for the manufacture of vinegar, but yields a product of very inferior quality, because on the wine being subjected to acetic fermentation the lactic acid contained in it is readily converted into butyric acid, which possesses a disagreeable rancid odor completely killing the pleasant aroma of the bouquet substances. There only remains as a material actually fit for the preparation of wine-vinegar, wine attacked by "acetic degeneration," *i. e.*, wine already so much changed by the vinegar ferment as to render it unfit for a beverage, and, further, wine which though not sick is unsound, showing a taste of mold, of the barrel, etc.

Wine no longer young and not overly rich in alcohol is especially adapted for the nutriment of the vinegar ferment. Such wine need only be exposed to a somewhat higher temperature in order to induce acetic fermentation, which if not disturbed in its progress, will finally convert all the alcohol in the wine to acetic acid.

It may be here remarked that every normal wine always contains, besides the bodies belonging to the series of fatty acids, acetic acid, though only about a few ten-thousandths of its weight. By storing the wine, the acetic acid does not increase, but becomes rather less, it being consumed in the formation of compound ethers. Hence, a rapid increase of the acetic acid is an indication of the wine being attacked by acetic degeneration, and if examined with the microscope the ferment characteristic of acetic fermentation will be found upon its surface. Many remedies have been proposed for the cure of acetic degeneration, but none of them is of any value except heating the wine to about 140° F., whereby the vinegar ferment is killed and the further progress of acetic fermentation checked. There is, however, absolutely no remedy for the removal or neutralization of the acetic acid already present in the wine. Heating the wine can only be recommended when the evil has been in existence but a short time and the increase of acetic



acid can be detected only by a very sensitive tongue. Mixing wine attacked by acetic degeneration with sound wine in order to cover the acid taste is especially unadvisable, since nothing can be attained by it except a short delay in the reappearance of the evil and the transmission of the infection to the sound wine. There are but two ways in which wine attacked by acetic degeneration can be in any wise profitably utilized: By employing it for the preparation of cognac or converting it into wine-vinegar. For the first a distilling apparatus is required, and, consequently, cannot be effected by every wine grower, while for the latter nothing is necessary but a few vessels readily procured.

Young wine attacked by acetic degeneration is also fit for nothing else than the preparation of vinegar. On account of its large content of albuminous substances it is, however, more suitable for the nutriment of the mold ferment than for that of the vinegar ferment, and consequently many difficulties occur in its conversion into vinegar. These difficulties can, however, be largely overcome by introducing large quantities of air into such wine and storing for some time in barrels filled up to the bung, or heating after the introduction of air to about 140° F., the separation of the albuminous substances being effected by either means, though more rapidly by the latter. Before further working the wine has to be filtered to remove the albuminous substances rendered insoluble by the treatment described, since their presence might give rise to injurious complications.

The so-called after wine obtained from grapes once pressed, by a process introduced by Petiot, is a very suitable material for making wine vinegar, since after fermentation its composition as regards alcohol and extractive substances is very advantageous. For successfully carrying out the process it is absolutely necessary to work the marc fresh from the press.

According to Paul Hassack \* there are used for the purpose,

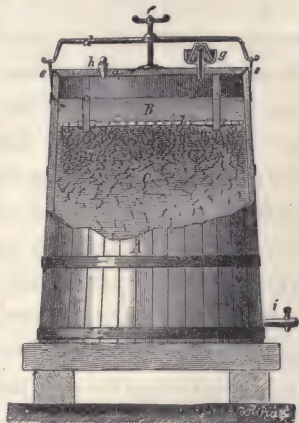
\* Gährungs-Essig, 1904.

400 lbs. fresh marc,\* 150 lbs. glucose and 1000 quarts of water.

The glucose is dissolved in about 400 quarts of hot water, and after making the whole up to 1000 quarts by the addition of 600 quarts of cold water, the solution is thoroughly stirred.

In the meanwhile 400 lbs. of fresh marc are brought into a fermentation-vat Fig. 45, and the sugar solution at a temperature of  $72^{\circ}$  to  $75^{\circ}$  F. is poured over them. The marc should be broken up, and not brought into the vat in large lumps. Fermentation as a rule commences after six hours and as by it the marc is forced to the surface, the vat, as will be seen from the illustration, is furnished with a perforated head *B*, by which the marc is kept below the level of the liquid. Fermentation is effected under exclusion of the air, a hydraulic ventilating bung *g* being used, which permits the escape of carbonic acid but does not allow air to enter. Fermentation is best carried on at a temperature of  $68^{\circ}$  to  $75^{\circ}$  F., and the fermenting liquid is allowed to remain in the vat till but a very weak current of carbonic acid escapes through the ventilating bung (about 6 to 8 days). The young wine is then as quickly as possible racked off into barrels for the second or after-fermentation. In filling the barrels it is advisable to pass the wine through a medium fine hair-sieve. The wine should not come in contact with the air any more than can possibly be avoided to prevent it from acquiring a darker color. The marc is taken from the fermentation-vat and pressed.

FIG. 45.



\* An addition of 1 lb. or more of crushed quinces per 100 lbs. of marc imparts to the wine and indirectly to the vinegar a very agreeable aroma, and the large content of tannin effects a more rapid clearing of the wine.

An addition of tartaric acid to wine prepared by the above-described process is seldom required, and in any case a solution of crystallized acid per 100 quarts of the wine when racked off for the second fermentation will be sufficient. The treatment of the young wine in the cellar is the same as that of original wine. The barrels should be kept full up to the bung. During the first two weeks of the second fermentation it is of advantage to have a ventilating bung in each barrel. By taking into consideration the sugar solution that has been added to the marc and the sugar contained in the latter itself, the wine, when fermentation is finished, will contain about 7 to 8 per cent. alcohol, 5 to 10 per cent. acid, 0.5 per cent. sugar and 1.6 to 2.3 per cent. extract free from sugar, and would yield vinegar with 5.6 to 6.5 per cent. acetic acid.

The wine thus obtained when carefully made is dark yellow, has an agreeably pure taste, and its odor is aromatic and rich in bouquet. When made with the exclusion of air, it is durable, easily managed, and clarifies readily. After racking off several times into clean, slightly sulphured barrels, it is clarified by means of isinglass solution or filtering through a linen or paper filter. For fining with isinglass  $\frac{1}{2}$  to  $\frac{3}{4}$  oz. of isinglass is sufficient for each 100 gallons of wine. Pound the isinglass, cut it into small pieces and soak it for 12 to 24 hours in fresh water. When taken from the water, squeeze it thoroughly and bring it into a vessel together with 1 gallon of water in which  $\frac{3}{4}$  oz. of tartaric acid has previously been dissolved. The isinglass swells up and is converted into a thin jelly-like mass. Dilute this solution with 40 gallons of wine, add it to the wine to be fined and stir thoroughly. Very young turbid after-wine is not fit for making wine vinegar. After-wine for vinegar making should be perfectly bright, and at least 4 to 6 months old.

Before entering upon a description of the various methods of making wine vinegar it may be mentioned that a product of actually fine quality can only be obtained by a *slow* process of acetification, wine treated by the quick process yielding a product very poor in bouquet.



*The oldest method for making wine vinegar* is that to which the term "boiling of wine vinegar" (Weinessig Siederei) has been applied. A barrel was filled  $\frac{3}{4}$  full with wine to be converted into vinegar; a portion of the fluid was then heated to boiling and poured back into the barrel. Upon the wine thus heated to about 86° F., the development of the vinegar ferment commenced, and in the course of a few months the greater portion of the alcohol was converted into acetic acid. The greater portion of the contents of the barrel was then drawn off as "ripe wine vinegar," the barrel again filled  $\frac{3}{4}$  full with wine, and a portion of this heated. The operation was continued in this manner until so much slimy sediment had accumulated in the barrel as to render it necessary to entirely empty and clean it. This crude process, which, as mentioned, was known in Germany as "vinegar boiling," was similar to the method formerly in general use in France, and which, being still partially practised there in some large wine-vinegar factories, for instance in Orleans, may be designated as the

*Orleans or old French Process of Making Wine Vinegar.* The casks, called *mothers*, which are employed, hold not more than 22 gallons, each cask being filled  $\frac{4}{5}$  full. Immediately above the level of the fluid a hole is bored in the surface of the front end of each cask, this hole as well as the bung-hole remaining open; a stop-cock for the discharge of the fluid is placed in the lower part of the cask. The casks are placed in rows in the open air, eight, ten, fifteen, or twenty such rows constituting what is termed a *vinegar field*. This so-called fielding, which is carried on from spring to fall, may be suitable for the southern part of France, but cannot be recommended for more northern regions, as the temperature may fall very low during the night and rise very high during the day. Experience has shown that the propagation and efficacy of the ferments are very much injured by great variations of temperature, and consequently it is decidedly preferable to keep the casks in a room the temperature of which can be maintained

at, at least 68° F. The wine remains in these casks until it is converted into vinegar. The latter is then drawn off by means of the above-mentioned stop-cock and the casks are again filled with wine, etc. The hole in the front end of the cask and the bung-hole permit the free access of air to the surface of the wine. In other French factories the work is carried on according to a method somewhat different from the one just described. Casks having a capacity of up to 100 gallons are used, each cask having in the surface of the front end a square aperture, which serves to charge the casks with wine as well as for the entrance of air. The casks are placed in three rows one above another in a room which can be heated. In the beginning of the operation a certain quantity of strong vinegar is brought into the casks; about one-fourth of its volume of wine is then added, and at intervals of eight days about 10 quarts more. When the cask is nearly filled up to the above-mentioned aperture, the regular process of drawing off vinegar and filling up again with wine is commenced. If, for instance, 10 quarts of finished vinegar are drawn off, the same quantity of wine is replaced in the cask, and suppose that, according to the manner of working, 7, 8, or 10 days are required for the conversion of this quantity into vinegar, 10 quarts of vinegar are again drawn off after the expiration of that time, this being continued until a disturbance occurs.

In the course of time large masses of slimy matter consisting of albuminous substances, vinegar ferment vegetating below the surface (the so-called mother of vinegar), decayed vinegar ferment, etc., form a deposit in the cask, and finally accumulate to such an extent as to occupy half the volume of the cask, so that the latter has to be emptied and thoroughly cleansed. Sometimes the operation has to be interrupted much sooner on account of the contents of the cask acquiring a disagreeable, putrid odor. The appearance of putrefaction is generally due to vinegar eels settling in the interior of the cask—as a rule, immediately above the level of the fluid—and developing to such an extent that they form a slimy coat-

ing on the cask and upon the fluid and suppress the development of the vinegar ferment. These animalcules are destroyed by being deprived of air, and, hence, when the vinegar ferment is brought to vigorous development it withdraws so much of the oxygen from the air in the cask that many of them die and their bodies sink to the bottom, where they sooner or later putrefy. If this putrefying process takes place before a cleansing of the casks is considered necessary, it progresses to such an extent that the entire contents of the cask are converted into a stinking mass which has to be removed as quickly as possible. The casks in which such disturbances takes place must of course be carefully cleansed by sulphuring and washing with boiling water before they are again used.

The above-described method of making vinegar is full of defects. The presence of vinegar in a fluid which itself is to be converted into vinegar promotes, to be sure, the formation of acetic acid, but is not absolutely necessary, as has been frequently asserted, for the induction of the process. If such were the case, it would evidently be impossible for an alcoholic liquid, such as beer or wine to pass on its own account into acetic fermentation. The acetification of the casks with boiling vinegar is irrational, because by heating the vinegar and pouring it boiling hot into the casks, not only the vinegar ferment contained in it, but also that present in the cask or wine, is, if not absolutely killed, at least weakened to such an extent as to be incapable of converting alcohol into acetic acid. That acetic fermentation nevertheless takes place is very likely due to the following causes.

The hot fluid in the cask gradually cools off and is finally reduced to a degree of temperature most favorable to the development of the vinegar ferment; in the same proportion as cooling-off takes place the air contracts in the cask and air enters from the outside. The latter, however, carries with it germs of vinegar ferment which rapidly develop upon the fluid when reduced to the proper temperature and cause its acetification. The air penetrating into the cask may, how-



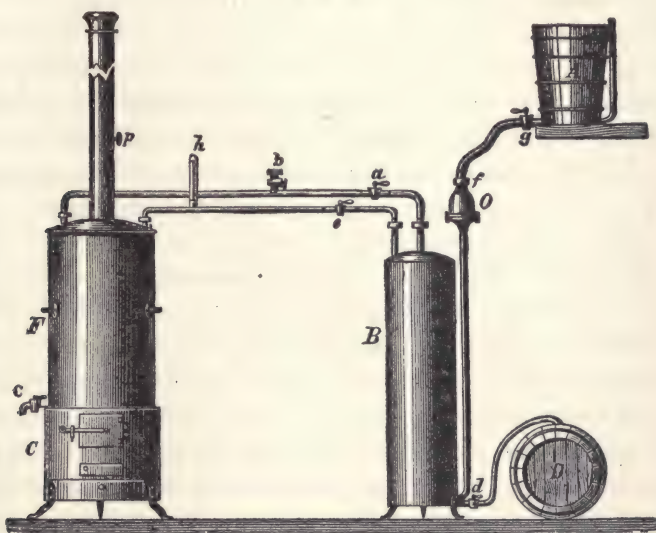
ever, accidentally contain no vinegar ferment, or that contained in it may not reach the wine; in such case the wine may for weeks remain in the cask without any perceptible acetification taking place until the latter finally appears by an accidental development of the vinegar ferment. This uncertainty can, however, be readily avoided by the direct culture by the vinegar ferment upon the wine to be acetified. Milk, as is well known, turns sour on exposure to the air by the milk sugar being converted into lactic acid by the action of a ferment frequently occurring in the air, this souring taking place in several hours or several days according to the temperature to which the milk is exposed. It is further a well-known fact that the addition of a few drops of sour to sweet milk suffices to immediately induce the formation of lactic acid in the latter; the ferment of lactic acid fermentation being in the true sense of the word sowed upon the milk. The ferment develops very rapidly, converts the sugar into lactic acid, and in a short time turns the entire quantity of milk sour.

Exactly the same course may be pursued as regards the vinegar ferment, it being only necessary to mix the wine with a fluid containing living vinegar ferment and place it in a sufficiently warm room in order to immediately start the process of the formation of acetic acid. In this case the vinegar ferment is sowed upon the wine, or in other words, the wine is impregnated with vinegar ferment and intentionally made "sick." This method of transmitting ferment to the fluid to be fermented has for a long time been in use in the preparation of beer and of alcohol. In the brewery the wort, and in the distillery, the mash, is brought into fermentation by "setting" it with yeast, *i. e.*, alcoholic ferment is intentionally added. The "setting of wine" with vinegar ferment is the only correct method for the preparation of vinegar from wine.

*Pasteur's, or Modern French Method of Preparing Wine Vinegar.* Pasteur, as previously mentioned, made exhaustive investigations regarding the conditions essential to the life of

the vinegar ferment, and found that it thrives especially well upon a liquid which in addition to water, alcohol and vinegar, contains a trace of phosphates. The latter is absolutely necessary for the propagation of the ferment; if wanting, the ferment cannot attain vigorous development. Pasteur recommends a liquid consisting of boiled water 100 per cent., pure alcohol 2, crystallized glacial acetic acid 1, phosphate  $\frac{1}{10}$ . As an inorganic combination that contains all the substances required for the nutriment and development of the vinegar ferment, Pasteur gives the following mixture of phosphate: Po-

FIG. 46.



tassium phosphate 1 part by weight, calcium phosphate 1, ammonium phosphate 2, magnesium phosphate 1.

The liquid prepared according to the above directions is exposed to the action of the air at a temperature of  $68^{\circ}$  to  $77^{\circ}$  F. In a short time the surface of the liquid becomes covered with the vinegar ferment and by the agency of the latter the alcohol present is converted into acetic acid. In the meanwhile a corresponding quantity of wine has been sterilized by

heating it to between  $158^{\circ}$  to  $176^{\circ}$  F. This process is called "Pasteurization" and may be effected in various ways, an apparatus for that purpose being shown in Fig. 46.\* Upon the furnace *C* sits a vessel *F* filled with boiling water. In this vessel lies a coil of pipe tinned, or better, silvered inside. A similar coil also tinned inside lies in the preparatory heater and cooler *B*. The wine to be heated is contained in the vat *A*. It passes through *g*, *f*, *o*, *d*, to *B*, and when the latter is full, passes through *e* into the coil in *F*, where it is heated, the temperature of the liquid being indicated by the thermometer *h*. From the coil in *F*, the pasteurized liquid is cooled by passing through *a* through the coil in *B*, heating at the same time the wine in *B*, and finally runs off at *d*. By regulating the cocks *b*, *e*, and *a*, the quantity of wine passing through the apparatus can be readily controlled so that the thermometer *h* constantly indicates a temperature between  $131^{\circ}$  and  $140^{\circ}$  F.

Various methods based on the researches of Pasteur have been devised, but before entering upon a description of the process, it will be necessary to discuss a few undesirable phenomena which may appear in the conversion of wine into vinegar. A thick white skin having the appearance of a ruffle may frequently form upon the surface of the wine to be acetified, the wine in this case becoming constantly poorer in alcohol, but does not show acidity. Sometimes the previously steady increase in the content of acid in the wine suddenly ceases and a very rapid decrease in the content of acid takes place, the development of the white skin upon the surface being also in this case observed.

The formation of this white coating upon the surface is due to the development of mold ferment which in a short time propagates to such an extent as to form a thick membranous layer, the folds being formed by the superposition of the cells. The mold ferment has the property of converting alcohol as well as acetic acid into carbonic acid and water, and conse-

\* Gährungs-Essig by Paul Hassack.



quently if it settles upon the wine the latter becomes poorer in alcohol, and if upon wine containing already a certain quantity of acetic acid the latter is also decomposed. The mold ferment requires, however, considerable quantities of nitrogenous combinations for its vigorous development, and therefore, readily settles upon *young* wine which contains a large quantity of albuminous bodies in solution. This fact explains the reason why young wine is seldom attacked by acetic degeneration, but it readily becomes moldy, and, consequently cannot be recommended as vinegar material except the albuminous substances be first separated by heating the wine to 140° F., which is best effected by means of the apparatus shown in Fig. 42.

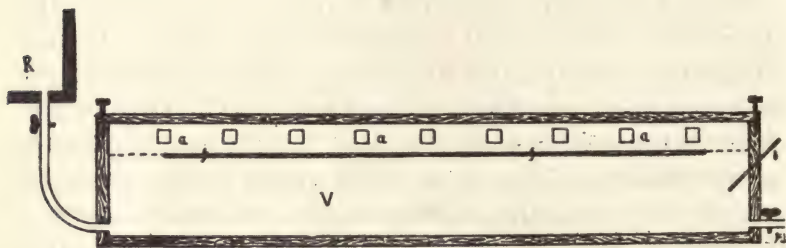
Another serious annoyance in making wine-vinegar is the appearance of vinegar eels, which, if not checked in time, may lead to the interruption of the entire process. These animalcules are seldom found in factories working with pump or well water, but frequently in those using river water, and consequently their introduction is likely due to such water. In case of their appearance in large masses it is best to interrupt the process in time in order to prevent the previously mentioned phenomena of putrefaction. The fluid containing the vinegar-eels should be drawn off into a thoroughly sulphured barrel. The sulphurous acid kills the vinegar eels as well as the vinegar ferment, and the filtered fluid, after standing a few weeks, whereby the sulphurous acid is converted into sulphuric acid, can again be used as alcoholic liquid. The vessels in which the vinegar eels have settled must also be thoroughly sulphured and then repeatedly washed with water before being re-used for making vinegar.

Throughout the entire factory the greatest cleanliness should prevail; in fact one cannot be too scrupulous in this respect, as otherwise by-fermentations readily take place, and another plague, the vinegar lice, or more correctly vinegar mites (see p. 140) may appear. Should either of these drawbacks happen, the workroom, fluids, and vessels should be thoroughly disinfected by means of sulphurous acid.

As previously mentioned the Orleans method of making wine-vinegar cannot be recommended, it being slow and laborious, and besides there is considerable loss of material by evaporation and by the formation of large masses of gelatinous "mother of vinegar," which depreciates the quality and necessitates expensive cleaning of the casks.

*Claudon's Method of Making Wine Vinegar.* This is one of the methods based on the researches of Pasteur. The apparatus used, Fig. 47 is described by Frederic T. Bioletti \* as follows: "It consists essentially of a wide, shallow, covered, rectangular vat, furnished with numerous openings near the top, *a* by which the entrance of air can be facilitated and

Fig. 47.



regulated. This vat is filled to near the bottom of the air vents with a mixture of 4 parts of good new wine and 6 parts of wine which has been pasteurized at 140° F., and when necessary filtered. On top of this liquid is floated a light wooden grating *f*, which helps to support the bacterial film and prevents its breaking and submerging during the various operations. When filled, the process is started by placing a small quantity of a good bacterial film on top of the liquid which soon becomes completely covered when the proper conditions of temperature and aeration are maintained.

"Each acetifying vat is connected with a small measuring vat *R* from which the proper amount of liquid is taken every

\* "Grape Vinegar." University of California Publications College of Agriculture, Agricultural Experiment Station. Bulletin No. 227, 1912.

day after a corresponding amount of vinegar has been removed. These two vats constitute a unit, several of which, usually six, are united in a battery. A factory includes several of these batteries.

“ The batteries are fed from a large vat or reservoir, where the mixture of wine and vinegar is prepared and stored. The vinegar drawn from the batteries runs directly to filters, from there to a pasteurizer, and thence to the storage casks. The output of these batteries is from two to five times as great per square yard of acetifying surface as that of the old methods; the cost of operation is considerably less, the loss by evaporation much reduced, and the quality equal and much more under the control of the manufacturer.”

*Bersch's Method of Making Wine-Vinegar.* The essential part of the entire process is the impregnation of the wine in suitable vessels with pure vinegar ferment under conditions suitable for the rapid propagation of the ferment. The vessels are so arranged that the finished vinegar can be removed and replaced by wine to be acetified without disturbing the ferment, one being thus enabled to uninterruptedly continue the process of the formation of vinegar for a long time, and producing vinegar unsurpassed by any other product as regards delicacy of taste and odor. According to the above statement, the operation includes the culture of the vinegar ferment on a small scale and on a large scale, the former for the production of pure ferment and the latter for obtaining wine-vinegar.

The culture of pure vinegar ferment on a small scale is best effected by heating wine in a porcelain or glass dish to between 140° and 150° F., then mixing it with an equal volume of vinegar and pouring the resulting fluid into shallow porcelain plates, which are placed in a warm room. In a short time, generally in 24 to 30 hours, the veil-like layer of vinegar ferment previously described is observed upon the surface of the fluid. If, besides the *dull* spots which are characteristic of pure vinegar ferment, spots of pure white color are formed, it is an indication of the development of mold ferment. The contents of



the plates showing this phenomenon have to be boiled and then again exposed to the air.

The wine to be acetified is in large, shallow vats, and is brought to fermentation by carefully submerging in it one of the above-mentioned plates containing pure vinegar ferment, so that the latter is distributed upon the surface; the plate is then withdrawn. The ferment propagates very rapidly, so that, in 24 hours, the surface of the wine in the vat is entirely covered with a thin veil of it. By keeping the temperature of the room in which the vats are placed at about 68° F., the acetification of the wine proceeds rapidly, tests repeated at intervals of 24 hours showing a constant increase in the content of acid, until in about 8 days all the wine is converted into vinegar when it is drawn off. To avoid the necessity of especially impregnating the next quantity of wine the finished vinegar is not entirely drawn off, a small quantity, (about  $\frac{3}{4}$  to an inch deep), upon the surface of which the vinegar ferment floats, being allowed to remain in the vat. By now introducing a fresh lot of wine the vinegar ferment propagates upon it and after some time converts it into vinegar.

With sufficient care the process of the formation of vinegar could thus be uninterruptedly carried on for any length of time by transferring the vinegar ferment from the finished vinegar to the wine, if a cleansing of the vat were not from time to time required, on account of the accumulation on the bottom of the vessel of decayed vinegar ferment and flakes of albumen which have become insoluble. When the vat is to be cleansed the last batch of vinegar is to be drawn off as long as it runs off clear, and the turbid remainder in the bottom of the vat is collected in a special cask, where it is allowed to clarify. The vat is then thoroughly cleansed with water, and after filling it again with wine, the latter is mixed with pure vinegar ferment in the manner already described.

If, as may happen in very rare cases, mold ferment in the form of the above-mentioned white spots appears upon the surface besides vinegar ferment, the vat must at once be

emptied. The process should also be interrupted in case of the development of the so-called mother of vinegar. The latter appears generally in the form of a soft gelatinous mass submerged in the fluid, and consists of vinegar ferment, which, however, on account of not being in direct contact with the air, does not produce acetic acid. The fluid to be acetified can be readily separated from the mother of vinegar by filtering through a close cloth, the mother of vinegar remaining upon the latter and finally drying to a whitish mass resembling very thin tissue paper.

From the above description it will be seen that the rational preparation of wine-vinegar is a very simple matter; but there are some difficulties which can, however, be entirely prevented or readily overcome. The vinegar ferment is very sensitive towards sudden changes in the composition of the fluid upon which it lives, as well as towards rapid changes in the temperature. The sudden change in the composition of the fluid is prevented by not drawing off all the finished vinegar, but allowing a small portion of it to remain in the vat. The fresh supply of wine entering from below then lifts up the remainder of vinegar, together with the ferment floating upon it, and the mixture of both fluids is effected so gradually that the change in the composition of the nourishing fluid proceeds very slowly. A sudden change in the temperature of the work-room can, of course, be readily prevented by proper heating.

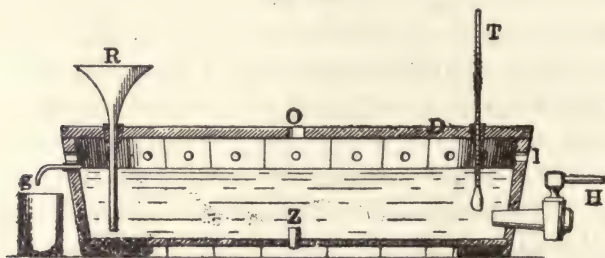
Ripe wines with not much above 6 per cent. of alcohol are the best to use, as they yield vinegar with about  $5\frac{1}{2}$  per cent. of acetic acid. Stronger wines with a content of alcohol up to 10 per cent., are, however, best reduced to about 6 per cent., either by water or ordinary vinegar. The strength of the latter must be so chosen that the wine-vinegar prepared from a mixture of wine and vinegar contains  $5\frac{1}{2}$  to 6 per cent. of acetic acid. The proportions in which vinegar and wine are to be mixed for this purpose are found by a simple calculation after an accurate determination of the content of alcohol in the wine and that of acetic acid in the vinegar.

The workroom should be so situated as to be protected against sudden changes in the temperature and provided with a furnace or self-regulating stove. The vessels for the formation of vinegar are placed upon suitable supports, and tables for holding the plates for the culture of the vinegar ferment should be provided. If the size of the room permit, it is advisable to store in it a few barrels of the material to be worked, the fluid thereby gradually acquiring the proper temperature.

For the formation of the vinegar very shallow vats, best with a diameter of  $3\frac{1}{2}$  to 5 feet and a depth of 9 to 14 inches, are used.

The iron hoops are protected from the action of the acid vapors by a coat of asphalt lacquer. The vats are placed in

FIG. 48.



the position they are to occupy in the workroom and filled with water up to about  $1\frac{3}{4}$  to  $3\frac{3}{4}$  inches from the top, the height of the level of the fluid being marked on the inside wall. At distances of  $3\frac{3}{4}$  inches apart, and  $5\frac{3}{4}$  in large vats, holes, *l*, Fig. 48 of 0.39 inch diameter are then bored in the wall of the vat. One hole, however, is bored in a place about 0.39 inch deeper than *l*, and in this hole is fitted a glass tube, *g*, bent at a right angle, under which is placed an ordinary tumbler. In the bottom of the vat is a tap-hole, *Z*, closed by a stopper.

If the vat be filled during the operation with wine, the latter can only rise until it begins to run off at *g*. The level of the fluid being but little below the holes *l*, an uninterrupted change



in the layer of air above the fluid takes place. A wooden spigot, *H*, is fitted in the vat about  $\frac{3}{4}$  to 1 inch above the bottom. In the centre of the lid *D*, which lies loosely upon the vat, is an aperture, *O*; in a second aperture a thermometer, *T*, is inserted, whose bulb dips into the fluid; and in a third aperture is fitted a glass funnel, *R*, reaching nearly to the bottom of the vat.

The operation in such a factory commences with the culture of the vinegar ferment. For this purpose as many shallow porcelain plates as there are vats are placed upon the table, and wine to the depth of  $\frac{1}{2}$  to  $\frac{3}{4}$  inch is poured in each. The room should be heated and kept at a temperature of  $86^{\circ}$  F. The manner of the development of the vinegar ferment upon the fluid in the plates as well as the precautions which have to be taken has already been described. In the commencement of the operation the culture of the ferment requires great attention, it being frequently disturbed by the development of mold ferment, but when the factory is once in proper working condition it is readily effected because the air of the workroom then contains a large quantity of the ferment, which rapidly propagates on coming in contact with a fluid favorable for its development.

The vats are charged by allowing the fluid to be converted into vinegar to run in until it begins to pass out through *g*. The impregnation with ferment is then effected by carefully emptying the contents of one of the plates upon the surface of the fluid, so that the greater portion remains floating upon it. Finally the lid is placed upon the vat and the latter left to itself.

The ferment soon covers the entire surface of the fluid in the vat, and the commencement of the process of oxidation is in a short time recognized by the rise of the thermometer dipping into the fluid. As long as the quantity of alcohol in the fluid is comparatively large, the process of the formation of acetic acid and the propagation of the ferment takes place very rapidly and the thermometer rises constantly; but with

an increase in the quantity of acetic acid these processes become slower, which is indicated by a fall in the temperature of the fluid. The energy of the process must, however, not be allowed to sink below a certain limit, care being taken to keep it up by raising the temperature of the workroom, but not higher than is absolutely necessary for the correct working, as otherwise there would be a loss of acetic acid or alcohol by evaporation.

The most convenient and business-like manner of operating a factory arranged as above described is to *simultaneously* charge all the vats with alcoholic liquid, it being then entirely in one's power to regulate the heating of the workroom according to the indications of the thermometer dipping into the fluid. If, for instance, the operation commences at 77° F., the thermometer will soon be observed to rise even if the temperature of the workroom remains unchanged. By the oxidation of the alcohol sufficient heat is liberated to increase the temperature of the fluid to above 95° F. It is, however, advisable not to allow it to rise above 86° or 90° F., as otherwise the losses by evaporation are too great. Hence, if the fluid reaches this limit of temperature the heating of the workroom is so regulated as to prevent a further rise of the thermometer, and a constant temperature is maintained for several days until it commences to fall almost simultaneously in all the vats. This fall in the temperature, as previously mentioned, is an indication of the fluid now containing a comparatively large amount of acetic acid and of the slow oxidation of the remaining alcohol. In order to maintain the most favorable conditions for the efficacy of the vinegar ferment and to smoothly and rapidly complete the process the workroom is now so heated as to show a constant temperature of 86° F. as long as the fluid remains in the vat.

Side by side with the observation of the statements of the thermometer a chemical examination of the fluid has to be carried on, this examination gaining in importance the further the formation of vinegar progresses. If the content of alcohol

in the wine to be worked is known, the test is up to a certain stage limited to the determination of the acetic acid, but if the process has so far advanced that the fluid contains scarcely 1 per cent. of alcohol, the latter has also to be determined by means of the ebullioscope which will be described later on. From this moment on the course of the process must be very carefully controlled, and interrupted when still 0.15 or at the utmost 0.2 per cent. of alcohol is present. This small amount of unchanged alcohol exerts a favorable effect upon the quality of the vinegar, acetic ether being formed from it and a corresponding quantity of acetic acid during the time the vinegar has to be stored.

The interruption of the process is best effected by separating the fluid from the layer of ferment floating upon it. The stop-cock, *H*, Fig. 48, is opened and left open as long as fluid runs out. A layer of vinegar about  $\frac{3}{4}$  to 1 inch deep upon which floats the vinegar ferment, remains in the vat, and the stop-cock being closed a fresh supply of alcoholic liquid is introduced through the funnel *R* until it begins to run out through *g*. The process then commences anew in the manner above described.

Theoretically unlimited quantities of wine could be converted into vinegar by means of such an apparatus, as the vinegar ferment which floats upon the fluid that remains in the vat, rapidly propagates upon the fresh supply of wine and converts it into vinegar. In practice an occasional short interruption of the process is, however, necessary. During the conversion of the wine the greater portion of albuminous substances held in solution in it separates as flakes, and, further, a portion of the vinegar ferment sinks below the level of the fluid and assumes the form of the flaky masses called mother of vinegar. The result after a number of operations is a slimy sediment, which finally accumulates to such an extent that it has to be removed. This is effected, after the finished vinegar is drawn off, by opening the tap-hole *Z*, and removing the slimy mass by means of a broom or crutch. The vat is then



thoroughly washed with water and can be immediately recharged with wine. The slimy mass is best collected in a tall vat and allowed to rest. In a few days it separates into two layers, the upper one consisting of quite clear vinegar which can be used for filling up storage-barrels, and the lower one of a thickly-fluid mass from which a certain quantity of vinegar can be obtained by filtration.

The vinegar drawn off from the vats is brought into storage barrels which are filled up to the bung and closed air-tight. The volume of the vinegar decreasing by cooling, the barrels must from time to time be examined and kept filled up to the bung-hole. While stored in the barrels the vinegar almost completely clarifies, and by carefully siphoning off the clear portion, it can be at once brought into commerce without further treatment. When a considerable quantity of slimy sediment has collected in the storage-barrels, it is drawn off and brought into the above-mentioned clarifying vat, or is clarified by filtration.

In case of disturbances in the production by the appearance of mold ferment or vinegar eels, the process once commenced must be carried through as well as possible, and then the entire operation interrupted for the purpose of thoroughly cleansing the vessels by washing with boiling water or steaming. Under no circumstances should it be attempted to continue working with vats infected with mold or vinegar eels, as it would only lead to a considerable loss of material, and the cleansing of the vessels which would have to be finally done, would be more difficult.

If the vinegar has been made from clear, ripe wine, it will generally come quite clear from the apparatus used for its production. Should it be turbid, as may sometimes happen, it has to be filtered through a bag or other filter as described on p. 156. The turbidity is caused by various substances separated during the formation of the vinegar. Vinegar is much more difficult to fine than wine and for this reason alone, only clarified, ripe wines should be used for its production. The

simplest method to clarify the vinegar is to store it for several weeks in a cool cellar in casks filled up to the bung. The greater portion of the vinegar can then be drawn off perfectly clear and only the last portion will require filtering.

The filtered vinegar is brought into clean casks and stored in a cool cellar. However, while thus stored it may sometimes depreciate in quality and strength by unfavorable conditions of temperature or handling, such depreciation being indicated by a change in the aroma, and the acid taste loses its sharpness and shows a peculiar insipidity. The cause of this alteration may be attributed to the decomposition of the tartaric and malic acids in the vinegar by a ferment. The only sure remedy for this and all other alterations is to sterilize the vinegar by heating to 140° F. The apparatus, Fig. 46, p. 197, for pasteurizing wine, or any other form of pasteurizer may be used for the purpose. The vinegar should come out of the pasteurizer cool and the storage-barrels should be completely filled, bunged tight and placed in a cool cellar.

Wine vinegar acquires its special aroma only by being stored for several months. French manufacturers store their best quality of it for at least one year before offering it for sale and, of course, charge a good price.

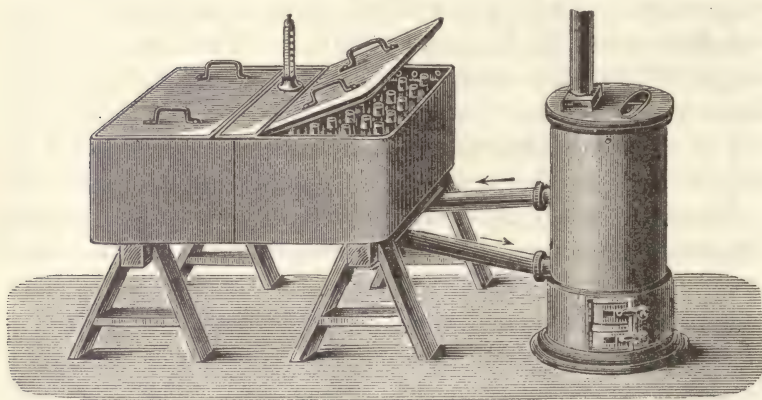
When the wine vinegar has acquired a fine taste and aroma by storing it should be bottled, this being the most profitable way of selling it. As the vinegar should be perfectly bright before racking it into bottles it must first be filtered or fined. Many manufacturers pasteurize the bottled vinegar. An apparatus for this purpose \* is shown in Fig. 49. It consists of an iron receptacle furnished with a cover fitting air-tight, and in the interior with a perforated false bottom. The apparatus is about 5 feet long, 3 feet 6 inches wide and 1½ feet deep. Between the perforated false bottom and the actual bottom is a pipe-system which is connected by two iron pipes with the furnace. The bottles are placed alongside each other upon the

\* German patent No. 17970 granted to Boldt & Vogel, Hamburg.

perforated false bottom. Before placing them in the apparatus, water is admitted into the latter so as to fill the space below the perforated false bottom and cover the latter. Fire is then started in the furnace and the water commencing to circulate, steam is in a short time evolved in the apparatus. The temperature prevailing in the interior is indicated by a thermometer on top of the apparatus, and pasteurization can thus be carried on to any desired degree. Cooling is effected by discontinuing heating and opening the cover.

Pasteurized wine-vinegar does not spoil if exposed to vary-

FIG. 49.



ing temperatures or even if kept in open or improperly closed receptacles. It is free from all kinds of ferments and vinegar eels, and is not attacked by the spores of the vinegar ferment suspended in the air, because the nutriment required for their further development has been withdrawn.

*Wine-Vinegar by the Quick Process.*—Although in making wine vinegar of fine quality, the best results are without doubt obtained by one of the slow methods above described, a very good product can be made from clear wines in one of the generators previously described, that furnished with a tilting



trough being by some manufacturers preferred for the purpose. The process is the same as for ordinary vinegar, the principal conditions for smooth working being a limited admission of air below the perforated false bottom, the use of perfectly clear, pasteurized wine, correct measuring of the quantity to be poured and its uniform distribution, absolute cleanliness, cleansing the perforated head every week or two, according to the accumulation of slime, and finally continuous working. The last condition—continuous working—is necessary to prevent the product from being impaired during the rest at night by a decomposition—a decrease of its most valuable properties.

Wine-vinegar made by the quick process has less aroma than that prepared by the other method. However, by storing it for about three months it gains in quality and aroma so that it can scarcely be distinguished from vinegar made by the Orleans method.

*Wine Vinegar from Marc*—The marc left after the wine has been pressed consists of the stems, skins and seeds of the grapes and contains a not unimportant quantity of must. As, previously described, by subjecting the marc with the addition of water or sugar solution to fermentation a wine is obtained which forms an excellent material for making vinegar. However, the marc may also be directly used for the purpose.

The mass of marc as it comes from the press is broken up and put in a pile where it is left to itself until it becomes warm and acquires the odor of alcohol and acetic ether. The mass is then shoveled into a vat and gently pressed together with a shovel. For every 220 lbs. of marc used, about 10 quarts of water are now sprinkled over the mass by means of a watering-pot. By the entrance of air while shoveling the pile of marc into the vat the action of the vinegar ferment has been accelerated and a considerable quantity of alcohol converted into acetic acid, which is indicated by the stronger vinegar odor. The water permeating the marc almost completely displaces the fluid containing the alcohol and acetic acid, the latter running off through an aperture in the bottom of the vat. It is

collected in a shallow vessel placed in an apartment having the ordinary temperature of a living-room, and is allowed to rest. The vinegar ferment present in abundance in the fluid rises to the surface, where it quickly propagates and converts the remainder of the alcohol in the fluid into acetic acid. The only difficulty to be overcome in preparing the vinegar according to this method is the appearance of the mold ferment upon the surface of the fluid. This can, however, be met by removing the growth of this ferment, which is recognized by its pure white color, by means of a spoon as soon as it has attained the thickness of a few millimeters. The vinegar ferment then commences to propagate and suppresses the further growth of the mold ferment.

If the grapes originally used contained from 18 to 20 per cent. of sugar, the vinegar from the marc prepared according to this method shows, if not too much water has been used, a content of at least 4 or 5 per cent. of acetic acid, and consequently is immediately fit for table use. By long storing in barrels kept filled up to the bung-holes, it acquires a flavor resembling that of vinegar prepared from wine.

On account of the simplicity and the slight expense connected with it, the above-described process is especially adapted for making vinegar for household use. For industrial purposes it is, however, more advantageous to prepare wine from the marc as described on p. 190, and convert the product thus obtained into vinegar.

## CHAPTER XIX.

## CHEMICAL EXAMINATION OF THE RAW MATERIALS AND CONTROL OF THE OPERATIONS IN A VINEGAR FACTORY.

*Determination of Sugar.*—The sacchariferous materials used by the vinegar manufacturer are either whiskey-mashes, malt-extracts, or must prepared from wine-marc, apples, etc. The determination of sugar contained in these fluids is effected by means of various instruments, which are really hydrometers, with different names and graduations. The instruments mostly used for the determination of sugar in whiskey-mashes and malt worts are known as saccharometers, and directly indicate the content of sugar in the fluid in per cent. A similar instrument, known as the must-aerometer, serves for the determination of the content of sugar in grape-must. According to the arrangement of their scales, the must-aerometers indicate either direct sugar per cent., or degrees; in the latter case the use of special tables accompanying the instrument is required for finding the per cent. of sugar corresponding to a certain number of degrees.

No special saccharometer for fruit-must having as yet been constructed, the determination of the content of sugar has to be effected either by a tedious method unsuitable for practice, or, what can be more quickly done, by fermenting a sample of the respective must, and after determining the quantity of alcohol, ascertaining from it the content of sugar.

In place of special saccharometers or must-aerometers, an ordinary areometer indicating the specific gravity can also be used, and the content of sugar corresponding to a certain specific gravity found from a reducing table. Tables X to XIII at the end of this volume give the content of sugar especially for wine-must, but also with sufficient accuracy for apple or pear-must, according to the statements of the respective must-aerometers.



*Determination of Alcohol.*—In a factory using commercial spirits of wine as the fundamental material for making vinegar, the percentage of absolute alcohol contained in it has to be accurately determined in order to enable one to correctly calculate, in the manner explained on p. 109, the quantity of water required for the preparation of alcoholic liquid of determined strength.

For the determination of the content of alcohol in pure spirits of wine consisting only of water and alcohol, instruments called *alcoholometers* are generally used, they indicating the volumes of alcohol contained in 100 volumes of the spirits of wine. They are, however, not suited for this purpose when, as is frequently the case in a vinegar factory, the spirit of wine contains other bodies besides water and alcohol. In this case, either the alcohol contained in a sample has to be distilled off, and after determining its strength by the alcoholometer, the content of alcohol in the total quantity of fluid ascertained by calculation, or the determination is effected in a short time and with sufficient accuracy for practical purposes by the use of a special apparatus.

*Determination of the Alcohol with the Alcoholometer.*—For the vinegar manufacturer the alcoholometer is an important instrument in so far as it serves for quickly ascertaining the degrees of the spirits of wine used. It is best to use an instrument which is combined with a thermometer, one being thus enabled to ascertain the temperature of the fluid simultaneously with reading off the statement of the alcoholometer. Tables I to VIII appended to this work give the necessary assistance for the determination of the actual content of alcohol in a fluid whose temperature is above or below the normal temperature (59° F.).

For examining fluids with a very small content of alcohol, alcoholometers have been constructed which accurately indicate at least 0.1 per cent. For the manufacture of vinegar, four alcoholometers will, as a rule, suffice. They should be so selected that one is to be used for fluids with from 0 to 4

per cent. of alcohol, the second for indicating 4 to 8 per cent., the third 8 to 12 per cent., and the fourth 12 to 16 per cent. The scale of such alcoholometers comprising only 4 per cent. each, is sufficiently large to allow of the easy reading off of one-tenth per cent. These instruments serve for the determination of the content of alcohol in alcoholic liquid consisting only of spirits of wine and water, and are used in examining the progress of the formation of vinegar during manufacture.

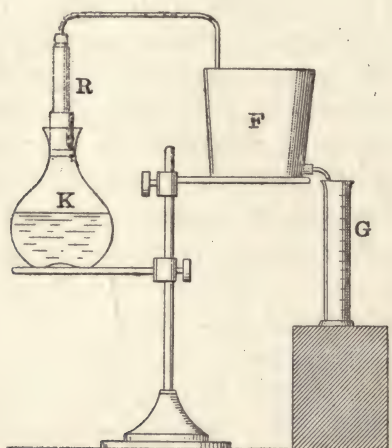
*Determination of the Alcohol by the Distilling Test.*—The content of alcohol in a fluid containing other bodies besides alcohol and water cannot be directly determined by means of the alcoholometer, as the statement of the latter would be incorrect on account of the foreign bodies exerting a considerable influence upon the specific gravity. Hence, the content of alcohol in alcoholic liquid containing a certain quantity of acetic acid, or fermented whiskey-mash, beer, wine, etc., cannot be ascertained by immersing the alcoholometer in the respective fluid. In order to determine the content of alcohol in such a fluid a determined volume of it is subjected to distillation, and the latter continued until it may be supposed that all the alcohol present is volatilized and again condensed in a suitable cooling apparatus. By diluting the fluid distilled over with sufficient water to restore it to the volume of the fluid originally used and immersing the alcoholometer the content of alcohol is determined.

A rapid and at the same time accurate execution of all examinations being of great importance in practice, a suitable apparatus should be used for the distilling test. Such an apparatus is shown in Fig. 50. It consists of a glass boiling flask, *K*, having a capacity of  $\frac{1}{2}$  liter in which sits by means of a perforated cork a glass tube, *R*, which is about  $\frac{3}{4}$  inch in diameter and  $7\frac{3}{4}$  inches in length. On top this tube is closed by a perforated cork. From the latter a glass tube bent twice at a right angle leads to a cooling-coil, which is placed in a vessel *F*, filled with water, and terminates over a graduated cylindrical glass vessel *G*. The uppermost mark on *G* indi-

cates the height to which the vessel must be filled to contain  $\frac{1}{2}$  liter = 500 cubic centimeters. Generally vessels are used which are so graduated that the distance between two marks is equal to  $\frac{1}{20}$  liter or 50 cubic centimeters. The boiling-flask stands upon a plate of thin sheet-iron (to prevent bursting from an immediate contact with the flame), and together with the cooling vessel is screwed to a suitable support.

In distilling a fluid containing acetic acid the vapors of the latter pass over together with those of alcohol and water, and consequently, the statement of the alcoholometer would be in-

FIG. 50.



correct. This is overcome by placing a few pieces of chalk the size of a hazelnut in the tube *R*. By the vapors coming in contact with the chalk the acetic acid is fixed to the lime contained in it, not a trace reaching the cooling vessel.

The manner of executing the test with this apparatus is as follows: Fill the vessel *G* to the uppermost mark with the fluid whose content of alcohol is to be examined, then pour it into the boiling flask *K*, rinse out *G* with water, and after pouring the rinsing water into *K*, put the apparatus together as shown in the illustration. The contents of *K* are then heated to boil-



ing by a spirit or gas flame under the sheet-iron plate upon which *K* rests, the flame being so regulated that the distillate flows in drops into *G*. By too strong heating the contents of *K* might foam up and pass into *G*, which would necessitate a repetition of the experiment with another quantity of fluid. Wine, beer, and whiskey-mashes frequently foam up on heating, which can, however, be almost completely overcome by the addition of a small quantity of tannin solution to the contents in *K*.

The heating of the boiling flask is continued until sufficient fluid is distilled over into *G* to fill it from one third to one half full, this being a sure indication of all the alcohol present in the fluid having passed over. The flame is then removed, the vessel *G* filled to the uppermost mark with distilled water, and the fluids intimately mixed by shaking, the mouth of *G* being closed by the hand. The fluid now contained in *G* consists only of water and alcohol, and its volume is equal to that of the fluid originally used. By testing the fluid with an alcoholometer the content of alcohol found corresponds exactly to that possessed by the fluid examined—alcoholic liquid, beer, fermented whiskey-mash, etc.

*Determination of the Alcohol by Means of the Ebullioscope.* Many determinations of the content of alcohol in the alcoholic fluid having to be made in a well-conducted vinegar factory, the above-described distilling test is objectionable on account of the time (about twenty minutes) required for its execution. Good results are, however, obtained by the use of the ebullioscope and, but a few minutes being required for the test with this apparatus, it can be frequently repeated, and thus even a more accurate idea of the working of the generators obtained than is possible with a single determination by the distilling test. The apparatus is very simple, is easily managed, and allows, without the use of an ærometer or table, of the direct reading-off of the content of alcohol in a fluid containing not much over 12 per cent. It is much used in France for the examination of wine. The principle of the apparatus is based

upon the initial boiling point of the fluid to be examined, an alcoholic fluid boiling at a lower temperature the more alcohol it contains. For instance, wine with—

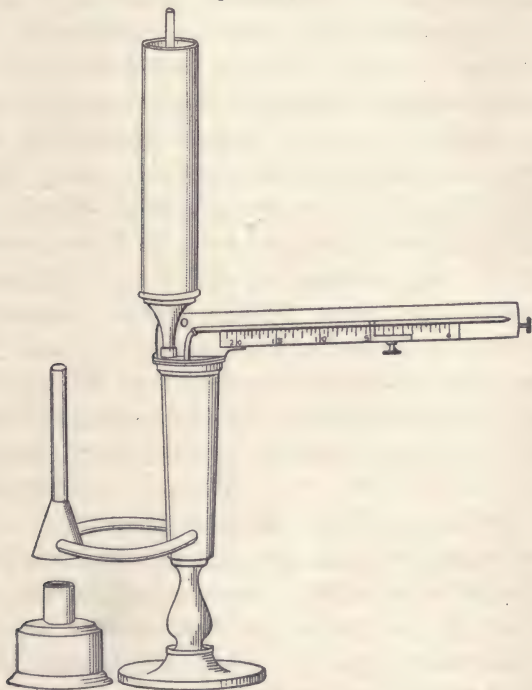
12 per cent. by volume of alcohol	boils at	196.7° F.
10    "            "            "	"            "	198.3° F.
8     "            "            "	"            "	201.0° F.
5     "            "            "	"            "	203.3° F.

Fig. 5I shows Vidal-Malligaud's ebullioscope. To a round cast-iron stand is screwed a thick-walled brass cup which expands somewhat towards the top; a screw-thread is cut in the upper edge. A hollow-brass ring is soldered into the cup near its base, the one end of the ring entering it somewhat higher than the other. On filling the cup with the fluid to be examined this hollow ring also becomes filled. On the one side the ring carries a small sheet-iron chimney, and by placing a small spirit lamp under this the fluid in the cup is heated, this arrangement securing a quick circulation of the fluid during heating. Upon the upper edge of the cup a lid is screwed, in which a thermometer is inserted air-tight. The mercury bulb of the thermometer is on the lower side of the lid and, in determining the boiling-point, dips into the fluid. The tube of the thermometer is bent at a right angle outside the lid, the latter carrying the scale, which is divided not into degrees but in per cent. by volume of alcohol. The scale can be shifted upon a supporting plate so that it can be fixed at any desired place, and, consequently, also so that the thermometer when dipped into boiling water indicates 0. The scale is secured by small screws. Into a second aperture in the lid is screwed the cooling-pipe, which is surrounded by a wide brass tube for the reception of the cooling water. During the determination of the alcohol, which requires about ten minutes, the cooling water need not be renewed, the boiling point remaining constant during the short time (one or two minutes) necessary for making the observation. In heating wine, the gases and besides a few light volatile varieties of

ether, as acetic ether, aldehyde, ethylamine, propylamine, and similar combinations escape through the cooling pipe, which is open on top, and in heating beer, carbonic acid. For the determination of the alcohol in sacchariferous wines, the ebullioscope is less adapted, nor does it give accurate results with the use of dilute wines.

It has been ascertained by the French Academy that the

FIG. 51.



statements of the ebullioscope as regards the quantity of alcohol in the wine differ on an average  $\frac{1}{6}$  per cent. from those found by accurate distillation. The entire apparatus with the exception of the thermometer being of metal, it is not liable to breakage. The mercury bulb of the thermometer is comparatively large. For the vinegar manufacturer the ebullioscope is a very valuable instrument, as it enables him to accurately



determine to within  $\frac{1}{5}$  per cent. the content of alcohol in a fluid in a shorter time than is possible with any other instrument. Its use is especially recommended when the working of one or more generators is to be ascertained in a short time, perfectly reliable results being obtained in connection with the determination of the acid by titration.

*Determination of the Content of Acetic Anhydride in Vinegar or Acetometry.* The content of acetic acid in vinegar is sometimes ascertained by a species of hydrometer termed an *acetometer*. The statements of these instruments are, however, very unreliable. Vinegar made from dilute alcohol or ripe wines in which no great excess of albuminous or other matter is present might to a certain limit be tested with sufficient accuracy by the acetometer, but vinegars made from malt, poor wines, and such liquids as contain an excess of organic matters, do not admit of being tested with the required degree of accuracy by this method, since the apparent quantity of real acetic acid is increased by the presence of foreign bodies which add to the density of the liquid. In some cases the vinegar is saturated with chalk or milk of lime, the solution filtered, and the specific gravity of the acetate of lime liquor ascertained, by which a nearer approximation is arrived at than by the direct testing of the vinegar, yet implicit reliance cannot be placed on either of these two methods.

The best method of ascertaining the percentage of acetic acid in vinegar is by titration or volumetric analysis. For the execution of the test a few instruments are required, namely, a burette and pipette. The latter is filled by dipping the lower end of it into the fluid and sucking on the upper end with the mouth until the fluid has ascended nearly to the top. The upper end is then quickly closed with the index finger of the right hand. By slightly lifting the finger the liquid is then allowed to flow off by drops until its level has reached a mark above the convex expansion of the instrument, when it will contain exactly the number of cubic centimeters indicated opposite to the mark.

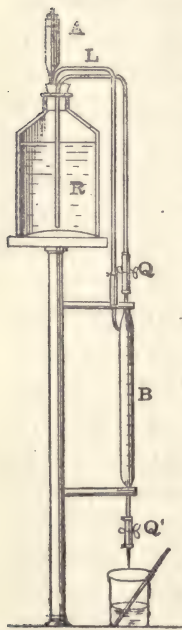
The burette is a cylindrical glass tube open on the top. It is graduated, commencing from the top, into whole, one-tenth and one-fifth cubic centimeters. The lower end of the tube is drawn out to a somewhat distended point so as to allow a rubber tube to be drawn over it and securely fastened. In the lower end a glass tube drawn out to a fine point is inserted. The rubber tube is compressed in the center by a pinch-cock or clip, whereby the lower end is closed. The burette is filled with fluid from above by means of a small funnel. By a quick, strong pressure upon the handle-joint of the clip, some liquid is then allowed to flow in a jet into a vessel. By this the tube below the clip is filled with liquid and the air contained in it expelled. By a slight or stronger pressure the liquid can, after some experience, be ejected in drops or in a stronger jet. The number of cubic centimeters which have been allowed to flow out can be readily read off by keeping the surface of the fluid in the tube on a level with the eye. The test liquor generally used is normal caustic soda solution, one cubic centimeter of it corresponding to 0.06 gramme of acetic anhydride, and for especially accurate determinations decinormal solution, one cubic centimeter of it corresponding to 0.006 gramme of acetic anhydride and  $\frac{1}{10}$  cubic centimeter to 0.0006 gramme.

For determining the acetic acid the burette is filled to the O point with soda solution. A corresponding quantity of vinegar is then accurately measured off by means of the pipette, and after bringing it into a beaker, colored red by the addition of one or two drops of litmus tincture and diluted with four or six times its quantity of distilled water. The beaker is placed upon a white support under the burette and the soda solution in the latter ejected in a strong jet by pressing with the right hand the handle-joint of the clip, the fluid being constantly agitated by gently swinging the beaker with the left. The inflow of soda solution is interrupted as soon as a *blue* coloration on the point where it runs in is observed. After thoroughly stirring the fluid with a glass rod, the soda solution is again allowed to

run in, but now drop by drop, the fluid being stirred after the addition of each drop. This is continued until the fluid has acquired a violet color with a strong reddish shade, and the addition of one drop more of soda solution changes the color to blue. The appearance of the violet coloration is called the *neutralizing point*, while the change of color from violet to blue indicates that the fluid is now neutral, *i. e.*, contains neither free acetic acid nor an excess of caustic soda. The determination is based upon the coloring substance of litmus appearing *red* in acid, *violet* in neutral, and *blue* in alkaline solutions.

Instead of soda test liquor, a solution of ammonia is sometimes used to saturate the acid. The solution is prepared by adding water to concentrated ammonia till the specific gravity is 0.992; 1000 grains of this dilute ammonia contain one equivalent of ammonia, which is capable of saturating one equivalent of acetic acid. The application of this test is similar to that already described.

FIG. 52.



There is some difficulty in preserving the dilute ammonia of the same strength, which is an objection to its use; but a uniformity of concentration may be insured by introducing into the bottle two glass hydrometer bulbs so adjusted that one remains barely touching at the bottom, and the other floats just under the surface of the liquid as long as the test-liquor retains the proper strength. If a part of the ammonia volatilizes, the specific gravity of the liquor will become proportionately greater, and the glass bulbs rise; the lower one higher from the bottom, and the upper one partly above the surface. When this happens, more strong ammonia is added, till the hydrostatic drops are properly readjusted.

Determinations of acetic acid by titration having to be frequently executed in a vinegar factory, it is advisable to



use an apparatus which will facilitate the operation. Such an apparatus is shown in Fig. 52. Upon a table stands a two-liter flask holding the normal soda solution. The flask is closed air-tight by a cork provided with three perforations. In one of these perforations is inserted a glass tube, *A*, in the lower end of which is a stopper of cotton upon which are placed small pieces of burnt lime. On top, the tube is closed by a glass tube drawn out to a fine point. Through another of these perforations passes a glass tube, *R*, bent twice at a right angle and reaching to the bottom of the flask. The portion of this tube outside of the flask, as will be seen in the illustration, is somewhat longer than that in the flask, and, consequently, the tube forms a siphon. The outside portion of this tube is connected by a short rubber tube with the upper portion of the burette *B*. The latter is secured in a vertical position by two rods placed on the stand holding the flask. Below the burette is connected with a short rubber tube in which is inserted a glass-tube drawn out to a fine point. On the side near the top of the burette is a small tube bent at a right angle, which is connected by a short rubber tube with the tube *L*, the latter reaching only to below the edge of the cork. Above and below the burette is closed by the clips *Q* and *Q*<sub>1</sub>.

For working with the apparatus the flask is filled with normal soda solution and the cork inserted air-tight after removing from it the tube *A*, and substituting for it a small glass-tube. Now open the upper clip *Q* and blow vigorously through the glass-tube substituted for *A*, whereby the fluid is forced through the tube *R* into the burette. This being done, cease to press upon *Q*, whereby the latter closes and stops a further discharge of the fluid. The tube, *A*, is then placed in position. By now pressing on the clip *Q* the fluid passes into the burette, the air contained in the latter entering the flask through the tube *L*. The burette being emptied by the discharge of the fluid through *Q*<sub>1</sub>, it is refilled for another determination of acid by simply pressing on *Q*, and this can be repeated as long as the flask contains soda solution.

In discharging the fluid from the burette by opening  $Q_1$ , air from the outside passes into the apparatus through  $A$ . In doing so it must, however, pass through the lime which fixes the carbonic acid contained in it, so that the fluid in the flask remains free from carbonic acid even after standing for months.

The calculation of the quantity of acetic acid present in the vinegar examined is made as shown by the following example:—

For 10 cubic centimeters of vinegar were consumed 70 cubic centimeters of decinormal soda solution.

One cubic centimeter of decinormal soda solution being equal to 0.006 gramme of acetic acid, hence 70 cubic centimeters=0.42 gramme.

Now, as 10 cubic centimeters contain 0.42 gramme of acetic acid, 100 cubic centimeters contain 10 times 0.42 gramme = 4.2 grammes of acetic acid; or the vinegar examined contains 4.2 per cent. by weight of acetic acid.

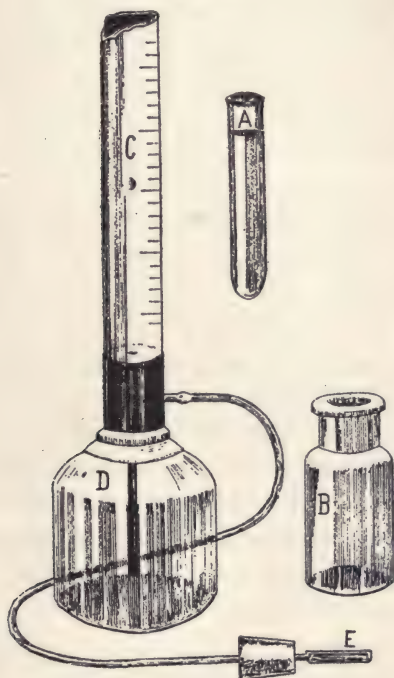
For the determination of the strength of vinegar with sufficient accuracy for manufacturing and commercial purposes an instrument called a *vinegar tester* is largely used. In the form shown in Fig. 53, as described by Frederic T. Bioletti,\* the acetic acid is determined by the volume of gas given off by bicarbonate of soda when treated with a measured volume of vinegar.

“The requisite volume of vinegar is measured in the small glass tube  $A$  and poured into the bottle  $B$ . A sufficient amount of bicarbonate is then taken with the spoon  $E$  and introduced carefully into the bottle. As soon as the bottle is tightly closed with the cork the bicarbonate is shaken gradually into the vinegar and immediately carbonic acid gas commences to be given off. This gas passing through the rubber tube forces the water in the bottle  $D$  to rise in the large glass tube  $C$ . The stronger the vinegar, the more gas will be given

\* Grape Vinegar. University of California Publications, College of Agriculture Experiment Station. Bulletin No. 227.

off and the higher the water will rise in the tube C. This tube is marked with numbered lines. By reading the number of the line nearest the level reached by the water and adding the estimated height above or below this line, the strength of the vinegar is obtained directly in per cent. If the vinegar is made from wine 0.5 per cent. must be deducted from the observed reading to allow for the tartaric acid of the wine.

FIG. 53.



“To insure sufficient accuracy with these instruments certain precautions are necessary. The bicarbonate of soda sold for cooking purposes is sufficiently pure. In placing it in the bottle care should be taken that none gets into the vinegar until the bottle is securely corked. There must be no leak in the apparatus. This is determined by allowing the column



of water to remain for a few minutes in the cylinder after making a determination. If the column does not fall in this time there is no leak of importance.

“The instruments are adjusted for water of ordinary temperature. If the water is either very cold or very warm the results are inaccurate. The following table shows some of the variations due to the use of too warm water.”

RESULTS OF VINEGAR TESTER COMPARED WITH ACCURATE ANALYSES

Vinegar.	True acidity.	Reading of vinegar tester.		
		65° F.	75° F.	86° F.
1 . . . . .	3.02	2.9	3.2	3.3
2 . . . . .	4.55	4.4	4.6	4.7
3 . . . . .	6.50	6.4	6.6	6.9
4 . . . . .	7.04	7.0	7.4	7.7
5 . . . . .	8.49	8.5	8.7	9.1
6 . . . . .	10.15	10.1	10.7	11.2

“No temperature correction is possible as the variations are irregular. At 65° F., as shown by the table, the determinations agree very closely with the results of more accurate tests. There are other sources of error such as the atmospheric pressure, the pressure of the column of water and the absorption of gas by the water, but they are none of them large enough to be of any significance to the vinegar maker.”

## CHAPTER XX.

## EXAMINATION OF VINEGAR AS TO THE PRESENCE OF FOREIGN ACIDS AND OF METALS, AS WELL AS TO ITS DERIVATION.

*Detection of Acids.*—Some unscrupulous manufacturers, in order to pass off weak or inferior vinegars, adulterate them with mineral acids. Such adulteration is not only a fraud, but dangerous to health, and it is necessary to indicate the means by which such additions can be detected.

*Sulphuric Acid.*—Add to a sample of the vinegar a few drops of a solution of barium chloride. If the vinegar becomes slightly cloudy, the impurities are due to sulphates naturally present in the water or in the substances from which the vinegar has been made. A heavy white cloud slow in subsiding will indicate free sulphuric acid in small proportion. If the quantity of sulphuric acid is more than a thousandth, the sulphate of baryta produces a precipitate and falls rapidly to the bottom of the test-glass.

The presence of free sulphuric acid in vinegar can also be determined by coating a porcelain plate with strong sugar solution and allowing the latter to dry up. By bringing a few drops of the vinegar to be examined upon the plate and placing the latter in a moderately warm place, pure vinegar evaporates, leaving a slightly brownish stain; vinegar containing free sulphuric acid leaves a dark-brown stain which on heating the plate turns black.

The presence of free sulphuric acid in vinegar can be determined with still greater sharpness by the following test: Divide a piece of starch the size of a grain of wheat in 50 cubic centimeters of vinegar and reduce the fluid one-half by boiling. To the clear fluid cooled to the ordinary temperature add a drop of a solution of iodine in spirits of wine. Vinegar containing no free sulphuric acid at once acquires a blue coloration; if free sulphuric acid be present, the fluid remains colorless.

This test is based upon the fact that starch by continued boiling with sulphuric acid is converted into dextrin and finally sugar. Neither of these bodies reacts upon iodine, while a very small quantity of starch gives with iodine the characteristic blue coloration.

*Hydrochloric Acid.*—Take about 100 cubic centimeters of the vinegar to be tested and distil off one-half by means of the apparatus Fig. 50, p. 216. Compound the fluid distilled off with a few drops of solution of nitrate of silver. In the presence of hydrochloric acid a white, caseous precipitate is immediately formed, which consists of chloride of silver and dissolves in liquid ammonia added in excess.

*Nitric acid* is not a frequent adulteration. It is detected by saturating with carbonate of sodium or of potassium several ounces of vinegar, and evaporating the whole to dryness. The addition of sulphuric acid and copper turnings will cause the evolution of nitrous vapors if nitric acid be present.

*Lactic Acid.*—In many varieties of vinegar small quantities of lactic acid occur, which can be detected by slowly evaporating 100 cubic centimeters of vinegar in a porcelain dish until but a few drops remain. If these drops show a very strong pure acid taste, the vinegar examined contains lactic acid. The presence of lactic acid is, however, not due to an intentional addition, but to the material used in the manufacture of the vinegar, that prepared from grain, malt or beer always containing it.

*Sulphurous Acid.*—This acid occurs only in vinegar prepared by fermentation when stored in freshly sulphured barrels. It may, however, occur in vinegar whose content of acetic acid has been increased by the addition of high-grade acetic acid prepared from wood-vinegar. The most simple method of detecting the presence of sulphurous acid is by placing 100 cubic centimeters of the vinegar to be examined in a glass distilling apparatus, and connecting the latter by a glass-tube with a vessel containing 50 cubic centimeters of



pure water compounded with about 10 drops of nitric acid. After distilling over  $\frac{1}{10}$  of the vinegar the acidulated water is heated to boiling for a few minutes and solution of barium chloride added. If the vinegar contains sulphurous acid, a heavy white precipitate is formed.

*Detection of Metals.*—The occurrence of metals in vinegar is due to the vessels employed in the manufacture or the storage, and, hence, the use of metallic utensils, such as stop-cocks, pumps, etc., should be avoided as much as possible. Besides iron, other metals such as copper, zinc and tin are occasionally found in vinegar.

*Iron.*—The presence of this metal imparts a black color to the vinegar, which is increased by a few drops of tincture of gall-nuts. If the color of vinegar compounded with a few drops of solution of tannin is not changed after standing a few hours, the vinegar contains no iron, or only so small a quantity as to be of no importance.

*Copper.*—While the presence of a small quantity of iron is of little importance in hygienic respect, that of copper, zinc, or tin is more serious, the combinations of these metals having a poisonous effect upon the organism. Copper in vinegar can be detected by evaporating to dryness about 1 quart of the vinegar to be examined and dissolving the residue in a few drops of nitric acid. By compounding a portion of this solution with ammonia in excess, the fluid acquires a perceptible blue coloration in the presence of copper. The latter can be shown with still greater sharpness by dipping polished iron into another portion of the fluid. If the iron becomes coated with a perceptible red film (consisting of actual copper), the presence of this metal is shown.

*Tin.*—Evaporate to dryness at least 2 or 3 quarts of the vinegar; dissolve the residue in hydrochloric acid, and conduct sulphuretted hydrogen through it until the fluid has acquired a strong odor of the latter. If a precipitate is formed, it is filtered off, dissolved in strong hydrochloric acid, and the solution divided into several portions. Compound one of these

portions with dilute solution of chloride of gold ; if after some time it becomes red and precipitates red flakes, the vinegar contains tin. The presence of tin is also indicated if another portion of the solution of the precipitate in hydrochloric acid does not acquire a blue color after the addition of potassium ferrocyanide. The behavior of the fluid towards solution of potassium permanganate may serve as a controlling test ; if the fluid contains tin, the solution of potassium permanganate becomes discolored.

*Determination of the Derivation of a Vinegar.*—The examination of a vinegar as regards the materials used in its preparation is generally effected by the senses of odor and taste. There are, however, a number of tests of ready execution which assist the judgment of the tongue and nose.

Vinegar prepared from dilute spirits of wine is colorless or only colored slightly yellowish. If such vinegar has a dark yellow color resembling that of wine, it is generally due to the addition of caramel, the addition being chiefly made on account of the erroneous opinion prevailing among the public that vinegar clear as water or only slightly colored lacks strength.

Vinegar prepared from spirits of wine, when carefully evaporated in a porcelain dish, leaves a very small residue of a whitish or very slightly yellow color, which chiefly consists of the salts contained in the water used for the preparation of the alcoholic liquid, an accurate examination showing it to consist of calcium acetate, gypsum, and a very small quantity of sodium chloride. If the residue is of a dark brown color, swells up when heated, and leaves a lustrous black coal, the vinegar has been colored with caramel.

Beer and malt vinegars are dark yellow, generally with a reddish shade. On account of their content of dextrin they foam when shaken, and, when carefully evaporated, leave a brown, gum-like residue. The latter consists chiefly of dextrin, and contains, besides the other extractive substances occurring in beer and malt vinegar, such as salts of ashes,

especially much phosphoric acid. On heating strongly an odor calling to mind that of toasted bread is evolved. At a still higher temperature the residue turns black and finally acts like caramel ; it evolves pungent vapors and leaves a lustrous coal.

The great content of phosphoric acid characteristic of malt or beer-vinegar may also serve for the determination of the derivation of such vinegar. By compounding beer or malt-vinegar with some nitric acid and a solution of ammonium molybdate and heating, the fluid, after standing, separates a yellow precipitate, which contains the phosphoric acid present in the fluid.

Wine-vinegar is best recognized by its characteristic odor, the latter becoming especially perceptible by rinsing out a large tumbler with the vinegar, and after allowing it to stand for a few hours examining the odor of the few drops remaining in the tumbler. The greater portion of the acetic acid having then volatilized, the vinous odor becomes more prominent. Cider-vinegar, the odor of which is somewhat similar to that of wine-vinegar, can in this manner be plainly distinguished from the latter, the residue in the tumbler having an entirely different odor.

The presence of potassium bitartrate is a characteristic sign of wine-vinegar. By evaporating wine-vinegar to a brownish syrupy mass, boiling the latter with some water, rapidly filtering the boiling fluid into a test tube, and adding double its volume of strong spirits of wine, a sand-like precipitate falls to the bottom of the test-tube, which consists of very small crystals of tartar. This, however, does not prove the sample to be genuine wine-vinegar, tartar also being contained in imitations. With a sufficiently sharp sense of smell this is, however the surest means of distinguishing genuine wine-vinegar from a spurious article.

In case the derivation of a vinegar is to be established with absolute certainty, it has to be subjected to an accurate chemical analysis, and this being better made by an analytical



chemist, only a few hints are here given which may serve as a guide for such analyses.

In vinegar prepared from a fermented fluid a certain quantity of glycerin and succinic acid will, as a rule, be present, these bodies being always formed by the fermentation of a sacchariferous fluid, and consequently when found the vinegar in question cannot have been prepared from an alcoholic liquid consisting only of spirits of wine and water. If they are found only in very small quantities, the alcoholic liquid used for the production of the vinegar consists very likely of spirits of wine and water with the addition of beer or fermented whiskey-mash, and in this case small quantities of dextrin and of phosphates will also be found. The total absence of tartaric acid and the presence of malic acid indicate the derivation of the vinegar under examination from fruit, though not necessarily from apples or pears, other sacchariferous fruits also containing malic acid. A content of tartaric acid is, however, no proof of genuine wine-vinegar, as its presence may be due to an intentional addition, and it is very difficult to arrive at a certain conclusion as to the genuineness of a pretended wine-vinegar, especially in the case of cider-vinegar to which tartaric acid has been added.

Should pepper, chillies, etc., be added to vinegar for the purpose of conferring more pungency, they may be detected by neutralizing the acid with carbonate of soda and tasting the liquor; if these bodies be present, the solution will still retain the sharpness peculiar to such spices.

## CHAPTER XXI.

WOOD VINEGAR AND OTHER BY-PRODUCTS OBTAINED IN THE  
DESTRUCTIVE DISTILLATION OF WOOD.

*Constitution of Wood.*—Wood essentially consists of woody fiber, small quantities of salts and sap and a varying quantity of hygroscopic water. Woody fiber or cellulose constitutes about 96 per cent. of dry wood, and is composed of  $C_6H_{10}O_5$ ; 100 parts containing 44.45 parts carbon, 6.17 hydrogen and 49.38 oxygen. The vegetable sap consists chiefly of water, but contains organic as well as inorganic matters, partly in solution and partly in suspension. The inorganic constituents—the ash left after the incineration of the wood—are the same in all kinds of wood. The quantity of water in wood is generally larger in the soft than in the hard varieties. One hundred parts of wood recently felled contain, according to Schübler and Neuffer, the following quantities of water:

Beech . . . . .	18.6	Common fir. . . . .	39.7
Birch. . . . .	30.8	Red Beech . . . . .	39.7
Oak . . . . .	34.7	Alder . . . . .	41.6
Oak ( <i>quercus pedunculata</i> ) .	35.4	Elm . . . . .	44.5
White fir. . . . .	37.1	Red fir. . . . .	45.2

The branches always contain more water than the trunk.

Wood is called air-dry when its weight no longer changes at an ordinary temperature; in this state it contains still 17 to 20 per cent. of water. The latter can be expelled by continued heating at 212° F., but wood thus dried re-absorbs about 20 per cent. of water from the air.

When felled, nearly all kinds of wood are specifically lighter than water. A few varieties are heavier, but these are the harder kinds in which the cellulose is so compactly packed as to leave very little space for the retention of air. The table here given exhibits approximately the specific gravity of the different woods:

Larch . . . . .	0.47	Ash . . . . .	0.64
Fir and pine . . . . .	0.55	Oak . . . . .	0.70
Beech . . . . .	0.59	Hornbeam . . . . .	0.76
Birch . . . . .	0.62		

The content of ash is not the same in all woods; it varying considerably in different parts of the same tree and also with its age. According to Violet, in the cherry tree the content of ash is greatest in the leaves (about 7 per cent.), next in the lower parts of the roots (5 per cent.); considerably greater in the bark than in the wood, in the former from 1.1 to 3.7 per cent., and in the latter 0.1 to 0.3 per cent. Saussure found in the bark of the oak 6 per cent., in the branches 0.4 per cent., and in the trunk 0.2 per cent. of ash. The ash consists chiefly of carbonates of calcium, potassium, and sodium, further of magnesia and the phosphates of different bases.

The average composition of 100 parts of air-dry wood is: Carbon 39.6 parts, hydrogen 4.8, oxygen and nitrogen 34.8, ash 0.8, hygroscopic water 20; and that of artificially dried wood: Carbon 49.5, hydrogen 6, oxygen and nitrogen 43.5, ash 1.

*Decomposition of wood.*—Cellulose when carefully treated remains unchanged for a long time, even thousands of years. Wood is, however, subject to greater changes, though under especially favorable circumstances it may last for several centuries. In the presence of sufficient moisture and air the nitrogenous bodies of the sap are, no doubt, first decomposed, and the decomposition being next transferred to the woody fiber, the latter loses its coherence, becomes gray, then brown, and finally decays. Hence, wood rich in water decays more rapidly than dry wood.

Wood to be preserved should, therefore, be as dry as possible, and the nitrogenous bodies, which can be but incompletely removed by lixiviation, be converted into insoluble combinations; tar and one of its most effective constituents—creosote—mercuric chloride, blue vitriol, chloride of zinc, and many other substances having been recommended for this



purpose. Moreover, it has been successfully attempted to produce certain insoluble bodies, such as aluminium and copper soaps, in the interior of the wood by saturating it with soda soap and then with aluminium chloride, or blue vitriol, or such as barium phosphate, by saturating with sodium phosphate and then with barium chloride, etc.

By heating to  $212^{\circ}$  F. wood remains unchanged, it yielding up only sap constituents. If, however, the temperature be increased, for instance to  $392^{\circ}$  F., a small quantity of sugar is, according to Mulder, formed from cellulose in a closed vessel, and from wood, according to G. Williams, an acid not yet thoroughly known, methyl alcohol (see further on), an oil boiling between  $277^{\circ}$  and  $421^{\circ}$  F., and a small quantity of furfural.

In the presence of water, wood in a closed vessel is, however, already decomposed at about  $293^{\circ}$  F. If this temperature be kept up for a long time, for instance, a month, the wood, according to Sorby, is converted into a lustrous black mass with the formation of acetic acid and gases.

According to Daubrèe, pine, when heated for some time with water in an entirely closed vessel to  $752^{\circ}$  F., is converted into a mass having the appearance of stone-coal and approaching anthracite in its behavior. Baroulier made similar observations, masses resembling stone-coal being formed by pressing saw-dust, stems and leaves together in moist clay and heating continuously to from  $392^{\circ}$  to  $572^{\circ}$  F., so that the vapors and gases could only escape very slowly.

By avoiding all heating, concentrated sulphuric acid converts cellulose into a gum-like body—dextrin—which by diluting with water and long digesting is converted into sugar (starch sugar). When heated, the wood, however, turns black and is completely destroyed, sulphurous acid being at the same time evolved. With concentrated sulphuric acid, cellulose swells up and gradually dissolves, being precipitated by water in white flocks. The starch-like body thus obtained is called *amyloid*. It is an altered cellulose and is colored blue by

iodine. At the ordinary temperature, wood is but little affected by dilute sulphuric acid, while at a higher temperature a certain quantity of sugar—glucose or dextrose—is formed, water being absorbed at the same time. This behavior has been utilized for obtaining alcohol by fermenting the sugar thus obtained with yeast after neutralizing the acid by calcium carbonate, for instance, chalk. The woody fiber remaining unattacked can be used as material for paper.

Concentrated hydrochloric acid colors wood rose color to violet-red and rapidly destroys it. Dilute hydrochloric acid on heating, forms sugar; but, according to Zetterlund, the quantity of absolute alcohol obtainable in this manner is very small, amounting to about 2.3 per cent. of the weight of the wood.\* By macerating wood with dilute hydrochloric acid at an ordinary temperature, the cellulose is not changed, but the so-called lignin seems to be dissolved. By forcing dilute hydrochloric acid by a pressure of two atmospheres into trunks provided with the bark, and subsequent washing out in the same manner with water, and drying by means of a current of air at 98.6° F., wood acquires great plasticity. In a moist state wood thus treated can be pressed together to one-tenth of its original volume.

Hydriodic acid reduces wood to various hydrocarbons, water being formed and iodine liberated.

Concentrated nitric acid, or, still better, a mixture of it and sulphuric acid, converts cellulose, for instance, cotton, into gun-cotton, while wood is colored yellow and partially dissolved. Dilute nitric acid, for instance, of 1.20 specific gravity, has no effect in the cold, and but little when heated.

By bringing cellulose in contact with dilute aqueous solutions of alkalies, it is colored blue by iodine, and consequently a starch-like substance is formed, but no humus-like bodies;

\* According to prior experiments by Bachet, it is, however, claimed that up to 23 per cent. of sugar can be obtained from wood by boiling 10 to 12 hours with water containing one-tenth of hydrochloric acid.

from wood only the lignin is extracted, the woody fibre remaining unchanged. By heating with strong alkaline lyes, or, still better, by fusing with solid caustic alkalies, acetic acid is, according to Braconnot, first formed and then oxalic acid. The latter acid is frequently obtained by this process.

On heating shavings with sodium sulphide an abundant quantity of acetic acid (sodium acetate) is formed, the addition of sulphur to caustic soda apparently having the effect of preventing the formation of oxalic acid.

*Products of Destructive Distillation.*—Besides charcoal, there are formed in the decomposition of wood under exclusion of air, a great number of products, the kind and quantity of which depend on the temperature to which the wood has been exposed, as well as on whether that temperature has been slowly raised to a certain point, or as rapidly as possible.

The products obtained by gradually increasing the heat are, at the ordinary temperature, either gaseous, liquid or solid. In speaking of the gases, which will be first considered, a distinction has to be made between those which must be accepted as actual products of decomposition of wood, and those which are formed by certain volatile fluids which are liquid at the ordinary temperature, but at higher degrees of heat suffer decomposition and yield gaseous products.

*Gaseous products of distillation.*—At the commencement of decomposition, between  $320^{\circ}$  and  $374^{\circ}$  F., carbonic acid,  $\text{CO}_2$ , mixed only with very small quantities of carbonic oxide,  $\text{CO}$ , is chiefly found, the quantity of the latter increasing with the rise in the temperature. At between  $392^{\circ}$  and  $428^{\circ}$  F. the quantities of carbonic acid and carbonic oxide are nearly equal, and small quantities of methane or marsh gas,  $\text{CH}_4$ , appear. At between  $608^{\circ}$  and  $680^{\circ}$  F., carbonic acid and carbonic oxide become less prominent, and methane appears in larger quantities. Above this temperature the content of carbonic acid in the gas mixture becomes small, while that of methane, mixed also with hydrogen, increases, and heavier hydrocarbons make their appearance.



By igniting the gases escaping from the distilling apparatus, a conclusion can be drawn from the appearance of the flame as to the kind of products which are developing. At first the flame is slightly luminous and shows the characteristic pale blue color of the carbonic acid flame. Later on, in consequence of the increase in the formation of methane and heavier hydrocarbons, the flame exhibits periodically a pure white color, while the blue coloration gradually becomes less prominent, until finally the gases burn with the luminous pure white flame of heavy hydrocarbons.

The following table shows the order in which the gaseous combinations are formed at different temperatures :

Temperature.	Name.	Composition.	
320° to 680° F.	Carbonic acid	$\text{CO}_2$	Gaseous at the ordinary temperature.
	Carbonic oxide	$\text{CO}$	
	Methane	$\text{CH}_4$	
	Hydrogen	$\text{H}$	
	Acetylene	$\text{C}_2\text{H}_2$	
From 680° to 812° F. (above this temperature only a very small quantity of gas is evolved).	Ethylene	$\text{C}_2\text{H}_4$	Liquid at the ordinary temperature.
	Propylene	$\text{C}_3\text{H}_6$	
	Butylene	$\text{C}_4\text{H}_8$	
	Benzene	$\text{C}_6\text{H}_6$	
	Toluene	$\text{C}_7\text{H}_8$	
	Xylene	$\text{C}_8\text{H}_{10}$	
	Cumene	$\text{C}_9\text{H}_{12}$	
	Napthalene	$\text{C}_{10}\text{H}_8$	

Pettenkofer found the composition of wood gases as follows :

	Air.	Carbonic acid.	Carbonic oxide.	Methane.	Hydrogen.	Heavy hydrocarbons.
Up to 680° F. . .	5	54.5	38.8	6.6	—	—
Above 680° F. . .	0	18-25	40-50	8-12	14-17	6.7

The process by which the above-mentioned bodies, of which the products of benzene caught by cooling may also be volatilized, are formed is of a very complicated nature, and while as regards the formation of many of these bodies only hypotheses can be advanced, that of others is readily explained.

Before entering upon this explanation, it is well to remember that in the execution of carbonization and destructive distillation on a large scale, it is impossible to maintain the same

temperature in all parts of the apparatus, and that considerable differences in temperature will occur. Furthermore certain volatile bodies when highly heated, *i. e.*, in contact with hot places of the distilling apparatus, possess the property of undergoing decomposition, new combinations being formed. This explains the origin of many bodies which appear among the products of the decomposition of wood.

When wood substance is heated, its elementary constituents act at first upon each other in such a manner that water is formed, and consequently steam is evolved, when the wood, after being freed from all moisture, is heated to  $320^{\circ}$  F. However, at a higher temperature the affinity of carbon for oxygen and hydrogen asserts itself, and at first combinations composed of the three constituents of wood are formed.

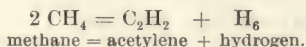
At a certain temperature the affinity of carbon for oxygen becomes so potent that the two bodies enter into combination, and carbonic acid—the combination of carbon richest in oxygen—is formed so long as an abundance of oxygen is present. At a later stage of decomposition, when the quantity of oxygen in the mass has decreased and the temperature has become higher, carbonic oxide—the combination of carbon poorer in oxygen—appears in larger quantity. It is due to the affinity of hydrogen for carbon that two hydrocarbons are formed. So long as hydrogen is present in abundance, methane  $\text{CH}_4$ —a combination of carbon with hydrogen completely saturated with hydrogen—is first formed, and then, at a somewhat higher temperature, ethylene  $\text{C}_2\text{H}_4$ , a combination poorer in hydrogen.

The gases above mentioned, namely : Carbonic acid, carbonic oxide, methane and ethylene, are very possibly those combinations which may originate directly from the decomposition of the wood substance. When these gases are brought in contact with glowing coal, or are highly heated—two cases which always occur in destructive distillation—they suffer decomposition, and new bodies appear in the products of distillation.

Carbonic acid in contact with glowing coal changes to carbonic oxide,  $\text{CO}_2 + \text{C} = 2\text{CO}$ , and it is very likely that the constant increase of the content of carbonic oxide in the gases with an increasing temperature, is partly due to this reciprocal action.

Hydrogen appears only when the temperature has reached a point at which methane and ethylene are formed in abundance, the hydrogen being split off from these combinations. So long as the temperature is not much above  $750^\circ \text{F.}$ , acetylene,  $\text{C}_2\text{H}_2$ , and hydrogen are chiefly formed from methane, while at a red heat, methane is directly decomposed to its elementary constituents.

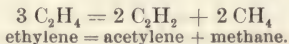
Hence at lower degrees of heat is formed :



and at higher degrees of heat :  $\text{CH}_4 = \text{C} + \text{H}_4$ .

On the portions of the iron distilling vessels, which are highly heated in the distillation of wood, a graphite-like layer of carbon is found which adheres quite firmly and is very likely formed by the decomposition of methane and other hydrocarbons in coming in contact with the hot surface, carbon being thereby separated.

Ethylene  $\text{C}_2\text{H}_4$  is decomposed at a comparatively low temperature to acetylene and methane.



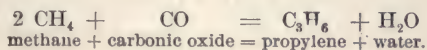
At a higher temperature, ethylene is decomposed so that the above mentioned products are formed, carbon, however being at the same time separated.



The above-mentioned decompositions of methane, however, are not the only ones which may occur, and according to the temperature, there may be formed from these gases a series of

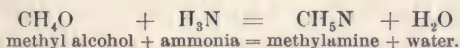


other combinations. The appearance of propylene may, for instance, be explained by the reciprocal action of methane and carbonic oxide.



Besides the processes of decomposition above represented, there are others which are a source of the formation of gaseous bodies. At a temperature of between 392° and 536° F. a considerable quantity of acetic acid and methyl alcohol is already formed, and the vapors of this combination are entirely or partially decomposed when highly heated, and this explains why in heating the wood very rapidly, very little acetic acid and methyl alcohol are formed, but, on the other hand, a very large quantity of gaseous and tar products.

Wood contains in its sap constituents small quantities of nitrogenous combinations and the nitrogen forms with hydrogen, ammonia, which, on coming together with hydrocarbons, forms at once substitution products, of which, for instance, methylamine, in which a portion of the hydrogen in the ammonia is replaced by methyl, appears in proportionally largest quantity.



Since in the destructive distillation of wood a large quantity of gases is evolved, the apparatuses in which the products of distillation, liquid at the ordinary temperature, are to be condensed must be quite large, as otherwise the seams might open in consequence of the pressure of the gases; or at least a large quantity of bodies which might be condensed would be carried away by the powerful current of gases.

By destructive distillation 100 kilogrammes of wood yield on an average 24.97 cubic meters of gas. This quantity, however, corresponds only to the conditions prevailing when distillation is carried on slowly. When the heat is rapidly raised

nearly 50 per cent. more of gas is obtained and of course the yield of liquid products of distillation and of charcoal is correspondingly reduced. F. Fischer made thorough investigations regarding the gases formed in the destructive distillation of wood. He found that the average yield from 100 parts of beech is 45 kilogrammes wood vinegar (with 4 kilogrammes acetic anhydride and 1.1 kilogramme wood spirit), 23 kilogrammes charcoal, 4 kilogrammes tar, 28 kilogrammes gases and steam.

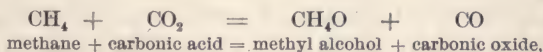
*Liquid Products of Distillation.*—The products of destructive distillation of wood which can be condensed by cooling, separate, when at rest, into two layers, the upper lighter one, which is of an acid nature, forming the wood vinegar, while the lower, denser one, is termed tar. Since these fluids are formed at temperatures widely apart and there is considerable difference in the chemical constitution of the bodies contained in them, it is best to consider them separately.

*Wood Vinegar.*—At the lowest temperature at which the decomposition of wood commences—according to Violette between  $302^{\circ}$  and  $312^{\circ}$  F., and according to Gillot, even between  $216.6^{\circ}$  and  $244.4^{\circ}$  F.—the three elementary constituents of wood act upon each other, and, besides a number of acids of the fatty acid series and methyl alcohol, there are formed certain products of the decomposition of these bodies. The formation of fatty acids, amongst which acetic acid appears in largest quantity, commences, according to Gillot, at  $255^{\circ}$  F., and reaches its maximum at  $437^{\circ}$  F. At higher temperatures considerable quantities of products of decomposition of the fatty acids appear, so that, according to the temperature and duration of distillation, there may be considerable variation, as regards the quantities of bodies contained in it, in the composition of wood vinegar gained on a large scale. The presence of the following fatty acids in wood vinegar has been definitely established :

Formic acid . . . . .	$\text{CH}_2\text{O}_2$
Acetic acid. . . . .	$\text{C}_2\text{H}_4\text{O}_2$
Propionic acid . . . . .	$\text{C}_3\text{H}_6\text{O}_2$
Butyric acid . . . . .	$\text{C}_4\text{H}_8\text{O}_2$
Valeric acid . . . . .	$\text{C}_5\text{H}_{10}\text{O}_2$
Caproic acid . . . . .	$\text{C}_6\text{H}_{12}\text{O}_2$

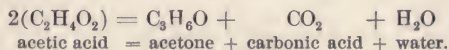
Formic acid boils at  $212^\circ \text{F.}$ , and since the boiling-point of the succeeding members of this series of acids lies about  $68^\circ$  higher, it may be supposed that the lower members of this series of acids are found in the portion of the wood-vinegar which distils over at a comparatively low temperature. This has been fully confirmed by experience, and for this reason the wood should be very slowly heated if the largest possible yield of acetic acid is to be obtained.

Methyl alcohol,  $\text{CH}_4\text{O}$ , may be produced from marsh-gas by subjecting that compound to the action of chlorine in sunshine, whereby chloromethane, or methyl chloride,  $\text{CH}_3\text{Cl}$ , is produced, and then distilling with potash. In the destructive distillation of wood it is very likely formed by the action of carbonic acid upon methane:



so that the appearance of constantly increasing quantities of carbonic oxide beside carbonic acid would appear to be explained by this process.

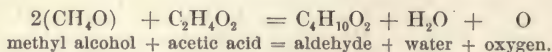
Acetone,  $\text{C}_3\text{H}_6\text{O}$ , is formed directly from acetic acid by conducting the vapors of the latter through a red-hot tube whereby carbonic acid and water are formed:



It may, however, be also formed from methyl alcohol and acetic acid, or from methyl alcohol and carbonic oxide. The occurrence of methyl acetic ether,  $\text{CH}_3\text{C}_2\text{H}_3\text{O}_2$ , in wood vinegar is due to acetic acid and methyl alcohol in a nascent state acting upon each other, while the presence of aldehyde,



$C_4H_{10}O_2$ , may be explained by the reciprocal action of methyl alcohol and acetic acid, two molecules of methyl alcohol with one molecule of acetic acid being transformed to dimethyl acetal (= aldehyde) while water and oxygen are liberated, the latter being immediately fixed by other products.

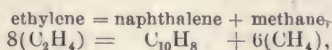


Further products of the reciprocal action of the fatty acids, methane and carbonic oxide, are: Metacetone,  $C_6H_{10}O$ , and allyl alcohol or furfurol,  $C_3H_6O$ . The small quantities of nitrogen originating from the sap constituents of the wood, which are present in destructive distillation, appear in the form of methylamine  $C_3H_2N$ , and ammonia.

There is such a variation in the quantities of the bodies of which wood vinegar is composed that it is impossible to give figures of general value in regard to them, the time during which heating takes place being of great influence in this respect, so that from the same variety of wood, by rapid heating, only a few tenths of the quantity of products are frequently obtained, which would result by slow heating. This fact is of the greatest importance for the practical manufacture of wood vinegar and wood spirit, and will be more fully discussed later on. It may here only be mentioned that from air dry wood, with not too rapid distillation, 30 to 53 per cent. (from most varieties of wood on an average 40 to 45 per cent.) of wood vinegar may be obtained, the specific gravity of which varies between 1.018 and 1.030, and which contains between 2.5 and 8.5 per cent. acetic anhydride, calculated from the gravity of the wood vinegar.

*Tar.* The products which occur in wood tar are still more numerous than those which originate during the period in which there is still considerable oxygen in the heated mass. Among the combinations which may be termed tar products in the actual sense of the word, are only a few containing oxygen, and these occur only in smaller quantities. The

larger quantity of the tar products consist of hydrocarbon combinations and must be considered as having been formed by the elements, carbon and hydrogen, grouping themselves in various ways at different temperatures. Ethylene very likely breaks up into a series of hydrocarbon combinations, at a slightly higher temperature than that at which it originates, as is, for instance, shown for naphthalene



and the formation of all the other hydrocarbons might in the same manner be explained. When derived from hard wood, wood-tar consists chiefly of paraffins,\* toluene, xylene, cresol, guaiacol, phenol, and methyl derivatives of pyrogallol.

Since a portion of these combinations is already formed at a temperature at which acetic acid is still evolved from the wood, certain quantities are found dissolved in wood vinegar, the tar products being jointly condensed with the wood-vinegar. While, according to Pettenkofer, the heavy hydrocarbons appear only at a temperature of above 680° F., according to Gil- lot, tar in abundance is already formed at 565° F. Practical experience, however, has shown that the largest quantities of tar are formed only at a higher temperature, and the boiling-points of tar products also indicate a high temperature of formation, naphthalene, for instance, boiling at 413.6° F. and paraffin only at over 572° F.

Besides the above-mentioned combinations, various chemists have established in wood-tar the presence of a long series of combinations, but up to the present time they have not been more closely examined, or they appear in such small quantities that their occurrence is only of theoretical interest. In this series may be mentioned: Iridol, citriol, rubidol, benzidol; further, retene, pittacall, cedriret, and pyroxanthogene; and

\* Under the name paraffin are very likely comprised many combinations which, though having the same percentage composition, possess different physical and chemical properties, *i. e.*, are isomeric.

further, the combinations established by Reichenbach, kapnomar, picamar, creosote and xylite, and finally mesite, prepared by Schweizer.

The yield of tar obtained in the destructive distillation of wood depends on the time and temperature used, but also essentially on the nature of the wood itself, resinous woods yielding larger quantities of tar than the varieties free from rosin. By slow distillation, the former yield 9 to 14 per cent., and the latter, 5 to 11 per cent. of tar. The more quickly the process of distillation is conducted the greater will be the yield of tar and gas, while that of acetic acid is less. The table below shows the bodies appearing in the destructive distillation of wood and the limits of temperature of technical importance, within which they are very likely formed:

GASES.		WOOD VINEGAR.		TAR.	
Carbonic acid . . . . .	320° to 680° F.	Formic acid . . . . .	356° to 572° F.	Benzene . . . . .	From 680° F. upward to red heat.
Carbonic oxide . . . . .		Acetic acid . . . . .		Toluene . . . . .	
Methane . . . . .		Propionic acid . . . . .		Xylene . . . . .	
Hydrogen . . . . .		Butyric acid . . . . .		Cumene . . . . .	
Acetylene . . . . .	680° to 809.6° F.	Valeric acid . . . . .	392° to 680° F.	Naphthalene . . . . .	
Ethylene . . . . .		Caproic acid . . . . .		Paraffin . . . . .	
Propylene . . . . .		Methyl alcohol . . . . .	392° to 680° F.	Phenol . . . . .	
Butylene . . . . .		Acetone . . . . .		Anisol . . . . .	
		Metacetone . . . . .	482° to 680° F.	Phenetol . . . . .	
		Acetic acid and methyl ether . . . . .			
		Dimethyl acetate . . . . .			
		Aldehyde . . . . .			
		Methylamine acetate . . . . .			

At present only a comparatively small number of the products originating in the destructive distillation of wood are utilized. Of the combinations which belong to the series of tar bodies, the only products which have actually found industrial application, are creosote, which consists chiefly of phenic acid, and the light and heavy oils which can be obtained by distillation from the tar.

It is, however, possible to separate from wood tar the various combinations contained in it, as has been successfully done with coal tar. Benzene, toluene, naphthalene, etc., can be prepared, and coloring matters manufactured from these hydrocarbons. The reason why this is not done is very likely to be sought in the fact that in the manufacture of illuminating gas



from coal, a sufficiently large quantity of coal tar is obtained to supply the demand of the manufacturers of coal tar colors. Paraffin might also be obtained from wood tar, but its manufacture would not pay in competition with the article obtained from the by-products gained in the purification of crude petroleum.

Hence there is nothing left in the destructive distillation but to work wood free from rosin into charcoal, acetic acid and methyl alcohol as chief products, and to consider all other products as by-products, to be utilized as opportunity offers and eventually to be used as fuel in the factory. When working with wood rich in rosin, it is best first to obtain acetic acid and wood spirit at a low temperature, and then to raise the heat to such a degree as is required to gain the total quantity of tar, since from this tar, by subjecting it to further treatment, tar oil can advantageously be prepared.

Wood-tar varies in character with the kind of wood from which it is obtained, that derived from resinous woods being considered the more valuable on account of its content of rosin. When wood is distilled in retorts, the portion of tar separated from the crude wood-vinegar by settling and that skimmed off of the top of the neutralized wood-vinegar are united, and, after washing with water, may be sold in the crude state as "raw tar" or as "retort tar." It is used for preserving wood, for making roofing felts, as an antiseptic, and for the preparation of wagon grease and other low-grade lubricants. In addition to the tar separated by settling, the crude wood-vinegar contains considerable tar held in solution by the acids and alcohol present, which is recovered when the wood-vinegar is distilled, and constitutes what is known as "boiled tar." It may be sold as such or burned under the retort, or it may be mixed up with the raw tar and subjected to any desired treatment.

By the destructive distillation of resinous woods tar oil containing turpentine is also obtained, but less wood vinegar, eventually calcium acetate. From 50 cubic meters of air-dry

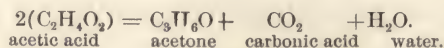
resinous wood are obtained about 4600 to 4750 kilogrammes charcoal, 1500 kilogrammes tar, 400 kilogrammes tar oil, 450 kilogrammes calcium acetate, 75 to 100 kilogrammes wood-alcohol.

*Properties of the Combinations formed in the Destructive Distillation of Wood.*—Of the many bodies formed in the destructive distillation of wood, only a few are of importance, these being especially acetic acid, wood spirit and the combinations and products of decomposition originating from these two bodies; further the combinations which can be obtained from the tar, namely: Creosote and tar oils.

*Acetic acid.*—The physical and chemical properties of acetic acid have already been described on p. 27. Of the products of decomposition which acetic acid may yield, those formed by the action of heat are here of special interest. Heated by itself, acetic acid can stand comparatively very high temperatures without suffering decomposition, and acetic acid vapor can, for instance, be conducted through a red-hot porcelain tube without being decomposed. However, decomposition takes place already at a slight red heat when the vapor comes in contact with glowing coal, as is the case in the destructive distillation of wood. If acetic acid vapor remains for some time in contact with such coal, a gas mixture, consisting of methane, carbonic acid and carbonic oxide is formed, the latter originating from the action of the coal upon the carbonic acid:



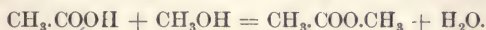
*Acetone.* At a less high temperature, acetone,  $\text{CH}_3\text{CO}.\text{CH}_3$ , is formed by the decomposition of acetic acid; its formation is illustrated by the following equation:



Acetone is always found in wood vinegar, and in a pure state is a liquid boiling at  $132.8^\circ \text{F.}$ , of specific gravity 0.814, of a

pleasant aromatic odor and a pungent taste. It burns with a brilliant flame and is miscible in all proportions with water, alcohol and ether. It is an excellent solvent for fats, resins, camphor, volatile oils, gun cotton, etc., and in modern times quite large quantities of it are employed in the manufacture of smokeless gunpowder.

*Methyl acetate* is in the chemical sense an acetic methyl ester. This ester is formed by the combination of acetic acid with methyl alcohol, water being withdrawn :



By treatment with alkalies such esters are saponified, whereby the alcohol and the salt of the acid are again formed. This is also the case with the methyl acetate contained in crude wood-vinegar when it is distilled with the addition of lime. But, as a rule, small quantities of it escape saponification, and for this reason the presence of acetic methyl ester can always be established in crude wood-vinegar.

*Aldehyde or acetaldehyde* has already been described on page 24. It is a constant constituent of the products of the destructive distillation of wood.

*Methyl alcohol or wood spirit*,  $\text{CH}_3\text{OH}$ , in a pure state, is a colorless, mobile liquid having a peculiar odor and burning taste. When ignited it burns with a slightly luminous flame. It boils at  $149^\circ \text{F}$ . and has a specific gravity of 0.798 at  $32^\circ \text{F}$ . It is a solvent for resins and essential oils, and for this reason is used in the manufacture of lacquers and varnishes. It may also be employed as fuel in place of ordinary alcohol. In modern times it has become an important article of commerce, it being largely used in the manufacture of aniline colors and for many other purposes.

Crude wood spirit as originally obtained from wood-vinegar is a mixture of methyl alcohol, acetic methyl ester, all the readily volatile products of decomposition of acetic acid (acetone, aldehyde, dimethyl acetate) and the readily volatile

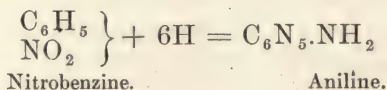


hydrocarbons. The preparation of pure methyl alcohol, which will be referred to later on, is therefore connected with certain difficulties.

*Tar Products—Hydrocarbons of the series  $C_nH_{2n-6}$ .* There is considerable variation in the properties of the products occurring in tar, as far as they are hydrocarbons. Those which are liquid at the ordinary temperature are of slight specific gravity but have different boiling-points and belong to different series composed according to a certain type. The best known are those of the series  $C_nH_{2n-6}$ , mentioned below.

Benzene boils at . . . .	179.6° F.	Specific gravity . . . .	0.850
Toluene     "     . . . .	231.8° F.	"     "     . . . .	0.870
Xylene     "     . . . .	282.2° F.	"     "     . . . .	0.875
Cumene     "     . . . .	330.8° F.	"     "     . . . .	0.887
Cymene     "     . . . .	345.2° F.	"     "     . . . .	0.850

These bodies possess the property of yielding by substitution bases which by treatment with oxidizing bodies can be converted into coloring matters, the so-called tar or aniline colors. As an example may here be mentioned the conversion of benzene. Benzene has the formula  $C_6H_6$ . By conversion of benzene into nitro-benzene =  $\left. \begin{matrix} C_6H_5 \\ NO_2 \end{matrix} \right\}$  and treatment of the latter with hydrogen at the nascent moment, aniline is formed :



According to the same scheme, nitro-combinations and amines can be prepared from toluene, cumene, and from all other hydrocarbons belonging to this series which can by suitable treatment be converted into coloring matters.

At present aniline colors are exclusively obtained from coal tar, but there is no doubt that they can also be prepared from wood tar, though thus far only experiments have been made

in this direction, which, however, have proved highly successful.

Besides the hydrocarbons, benzene, toluene, etc., boiling at a higher temperature, the presence in wood tar of a few others, distinguished by very low boiling points and slight specific gravity, has been established. Such are:

Iridol boils at	116.6° F.	Specific gravity	0.660
Citriol "	125.6° F.	" "	0.700
Rubidol "	134.6° F.	" "	0.750
Coridol "	140° F.	" "	0.800
Benzidol "	158° F.	" "	0.850

By treatment with nitric acid, these hydrocarbons can also be converted into nitro-combinations from which, by reduction, amines can be prepared, which may be converted into colored combinations. However, thus far these bodies have not been thoroughly examined.

*Naphthalene and Paraffin.*—These two hydrocarbons, which also occur in wood tar, are solid at the ordinary temperature and are distinguished by high boiling points. Naphthalene crystallizes in white rhombic leaflets with a peculiar odor and burning taste. It fuses at 174.2° F. and boils at 424.4° F. Its composition is  $C_{10}H_8$ . By treating with nitric acid, naphthalene may be converted into nitronaphthalene,  $C_{10}H_7NO_2$ , and from the latter naphthylamine is prepared, which serves for the manufacture of yellow and red coloring matters.

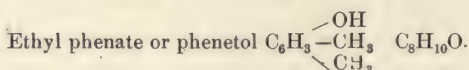
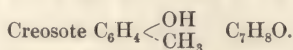
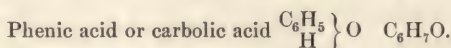
The hydrocarbons with boiling points of 680° to 752° F., which occur in wood tar, are designated paraffins and belong very likely to the series  $C_nH_{2n}$ . According to the material from which they are obtained, the melting points of the paraffins vary, and all the combinations belonging to this group are distinguished by their great chemical indifference.

Many varieties of wood tar are of a viscous and gritty nature, which indicates a large content of paraffin. In fact, paraffin was first prepared from beech-wood tar by Reichen-

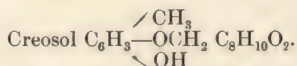
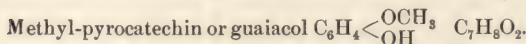
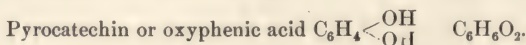
bach. However, as previously mentioned, there is at present little prospect of the preparation of paraffin from wood-tar proving remunerative, on account of the competition with the article obtained from crude petroleum.

Besides the hydrocarbons mentioned, there occur in wood-tar small quantities of the following : Chrysene,  $C_{12}H_8$ , retene,  $C_{18}H_{18}$ , and pyrene,  $C_{15}H_{10}$ .

*Tar Products Containing Oxygen (Creosote).*—The combinations containing oxygen which occur in wood-tar are either members of the phenol series or belong to the guaiacol series. The liquid known as wood-tar creosote consists of a mixture of combinations belonging to both series, the compounds belonging to the phenol series being :



Among these acids, carbolic acid occurs in smallest quantity. The combinations belonging to the guaiacol series, which are found in wood tar are :



Creosote may also be prepared from coal-tar ; chemically, however, this creosote consists almost exclusively of carbolic acid, and cannot be used for medicinal purposes.

Wood-tar creosote is a fluid of a peculiar penetrating smoky odor. Applied to a mucous membrane or to the raw cuticle, it excites severe burning pain, coagulates the albumen of the



secretion, and may even produce ulceration. It preserves meat probably in consequence of its behavior with albumen, and the preserving action of smoking meat is due to a content of this body in wood-smoke.

Since the introduction of the manufacture of illuminating gas from wood, wood-tar creosotes very rich in carbolic acid are found in commerce, and this may be explained by the conditions under which the tar from which the creosote has been obtained has been formed.

When illuminating gases are to be obtained from wood, the latter must as quickly as possible be heated to a high temperature, and under these conditions an abundance of carbolic acid is formed which passes into the creosote. If, on the other hand, destructive distillation is carried on at a slowly increasing temperature, tar is obtained which chiefly contains creosol and guaiacol. Since carbolic acid is comparatively quite a poisonous body, creosote intended for medicinal use should only be prepared at factories which carry on the destructive distillation of wood chiefly for the purpose of obtaining acetic acid.

Besides the above-described constituents, various chemists have mentioned a complete series of compounds as occurring in wood-tar. It can scarcely be doubted that several, but little known, bodies which have not yet been prepared in a pure state occur in wood-tar, but those which have been called eupioné, picamar, kapnomar, pittacal, cedrilet, pyroxanthogene, mesite, xylite, etc., consist very likely of mixtures of various bodies. As far as these compounds are known they must be considered as hydrocarbons belonging to various series of combinations. A few of them, like cedrilet and chrysene, give with acids characteristic color reactions, and pittacal is itself of a beautiful dark blue color. On heating it evolves ammonia, and hence it contains nitrogen very likely in the form of combinations known as substituted ammonias.

None of these combinations have thus far become of any technical importance, but they are occasionally observed in the purification of sodium acetate. The melt of the crude salt

when not yet sufficiently roasted yields, when treated with water, solutions of a beautiful blue, violet, red to orange color, these colorations being produced by the products of decomposition of the tar substances.

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## CHAPTER XXII.

### PREPARATION OF CHARCOAL, WOOD VINEGAR AND TAR IN CLOSED VESSELS.

IN installing a plant for the purpose of utilizing wood in a thermo-chemical way, *i. e.*, a factory in which chiefly pure acetic acid and wood spirit, and eventually the tar-products—tar oils and creosote—are to be produced, apparatus will evidently have to be selected, the arrangement of which is as complete as possible, and which, besides the gaining of all products liquid at the ordinary temperature, allows also of the utilization of the large quantities of gas evolved in the destructive distillation of wood. Hence, according to the object in view in distilling wood, the arrangements for obtaining the products of distillation may be of very varying nature, the most simple being a few suitably-fitted pipes and barrels. Retorts in which the temperature can be accurately regulated are the most complicated and complete, but also the most expensive, apparatus.

What kind of apparatus has to be used and what kind of products are to be prepared, depends entirely upon local conditions. Since the value of a product increases in proportion to the labor expended in the production of it, a manufacturer who prepares, besides pure acetic acid and wood spirit, also tar oils and creosote, will thus realize the greatest profit from the wood, but to attain this object the establishment of a plant with all the apparatus required for this purpose is necessary.

However, many a proprietor of woodland does not care to manufacture pure products in a special factory, but desires,

by the expenditure of a small sum, to obtain from his wood, besides charcoal, also wood-vinegar and eventually tar, in order to realize, by the sale of these raw products, a larger profit from his wood than is attainable by charring alone. In such a case the principle of division of labor, and eventually association deserves recommendation, so that the proprietors of the woodland would prepare charcoal besides wood-vinegar and tar and sell these products, or what is still better, work them further in a factory erected at joint expense.

Some readers may ask the question whether it is possible to utilize large quantities of wood by employing them for the manufacture of products of destructive distillation. This question may be unconditionally answered in the affirmative, since in consequence of the high and steadily increasing tax on alcohol levied in most countries, the price of table vinegar must constantly rise.

There are but two processes by which acetic acid on a large scale can be prepared, namely, from alcohol and from wood. Up to the present time alcohol can only be obtained from the products of agriculture, the chief raw materials being grain and potatoes, and eventually grapes and certain varieties of fruit, especially apples.

By taking into consideration the great expense of labor required to obtain these products of the soil and to manufacture alcohol and acetic acid from them, it will be evident that the price of this acetic acid must be greater than of that which can in a short time be prepared from wood. Furthermore, vinegar prepared from alcohol is never pure, *i. e.*, it does not consist only of acetic anhydride and water, but always contains quite a quantity of foreign substances in solution, and besides is very poor in acetic acid. To obtain the latter pure and in a highly concentrated state, vinegar has to undergo almost exactly the same chemical manipulations to which wood vinegar has to be subjected in order to prepare from it pure concentrated acetic acid, but the expense of preparing such acetic acid would be very great.



Pure concentrated acetic acid is at present used on an extensive scale in the chemical industries, and is also more and more employed in the preparation of table vinegar, since there is no difference between pure acetic acid prepared from wood and that prepared from alcohol. Wood vinegar, however, can be utilized for other purposes besides the preparation of pure acetic acid, it being in consequence of its content of tar products, which have an antiseptic effect, an excellent preservative of wood, and the process of impregnating the latter with it is decidedly cheaper than most methods in which other bodies are employed for the purpose.

When only acetic acid, wood spirit and tar are to be produced, wood waste of all kinds, for instance, saw-dust, exhausted shavings of dye-woods, spent tan, can be advantageously worked. If the charcoal obtained from such material is to be used as fuel, it must, on account of the smallness of the pieces, be moulded to briquettes by a machine constructed for the purpose.

*Kilns or Ovens and Retorts.*—The chief object of the oldest method of charring wood, as still carried on in some localities, is the production of charcoal without regard to the recovery of the available by-products. For this purpose the wood under a moveable covering is burnt with the access of air in heaps or pits in the immediate neighborhood of where it is cut. The attempt to obtain all the products simultaneously, with a greater amount of charcoal, probably first led to the substitution of stationary apparatus, either of brick-work or iron, in place of the covered heaps. Some of these arrangements are calculated, like the heaps, to produce the necessary temperature for charring, by the combustion of a portion of the wood, and the admission of a little air, such as kilns, the sides of which form a fixed covering for the substances to be charred. In others the portion of the wood destined to produce the heat is entirely separated from that to be charred, the latter being placed inside, the former outside, the kiln. The yield of acetate and alcohol is very low even in the best of kilns, and the

use of the latter has been almost entirely abandoned, they being now only employed in localities where charcoal is practically the only product recovered. For the sake of completeness a few of the older constructions will be described.

*Schwartz's Oven.*—The oven introduced by Schwartz into Sweden is based upon the principle that the hot gases furnished by a special furnace must pass through the wood piled in a closed space and heat it sufficiently for destructive distillation. The construction is shown in Figs. 54, 55 and 56. Fig. 54 is a ground plan, Fig. 55, a section of the elevation following the lines *dd*, and Fig. 56, another section following the lines *cc*. In the illustrations *AA* is the space where the wood is carbonized, *b*, apertures through which the wood is brought into the oven, and the charcoal withdrawn; *c c*, the fire-places for heating the oven; *d d*, openings through which the smoke, carbonic acid, acetic acid, oleaginous and tarry substances pass off through the pipes *g g*, and thence through the condensers into the chimney; *ee* are knee-pipes which convey the tar condensed into the vessels *ff*—Fig. 55. The bend in the pipes prevents the access of air into the apparatus. *H H H H* are wooden channels wherein the acid and oleaginous matters condense; *i* is the chimney and *k* a small opening in the chimney, where a fire is lighted to establish a powerful draught. The oven walls are of fire-brick or may be of two rows of ordinary brick, the interspace being filled with clay and sand. The oven is first charged with the heaviest blocks of wood, and between these smaller wood is introduced, for the purpose of making the interior more permeable to the action of the fire. All the orifices of the oven are then closed, and the fires at *c c* lighted, the current of air being instituted in *i* by lighting a fire at *k* as above mentioned. The blaze of the fire traverses the oven and carbonizes the charge of wood, and the smoke and other vapors from the oven pass by the exit pipes *d d* into *g g*, whence they escape to the condensers *H H*, and thence to the chimney *i*. The charge is known to be completely carbonized when the smoke issuing at *i*, which is at first black and heavy,

becomes bluish and light. The chimney passage is then closed, and the opening of the pipes *d d* stopped up with

FIG. 54.

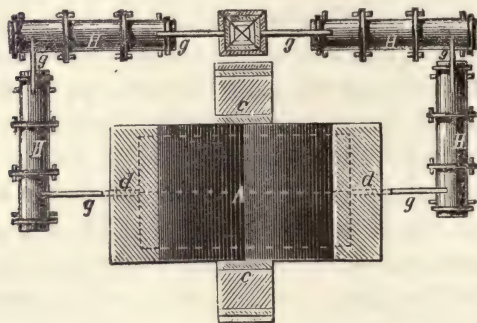


FIG. 55.

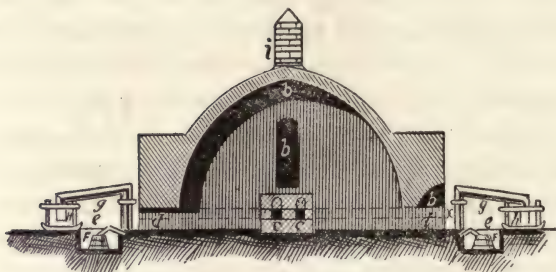
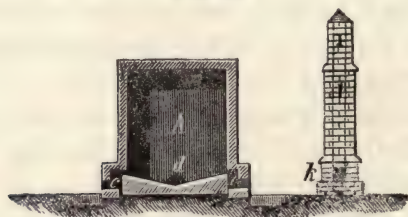


FIG. 56.



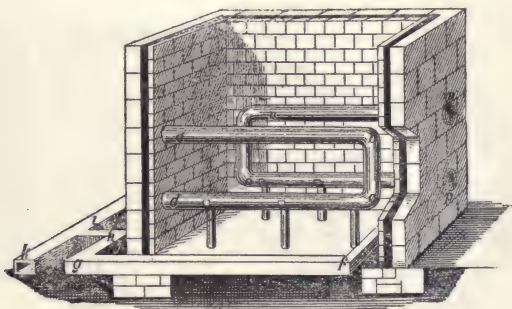
wooden plugs and then well luted with plastic clay; the fire-doors are closed and the oven left to cool. At the end of the second day, two holes in the top of the oven which hitherto



had been closed air-tight, are opened, and water is introduced to extinguish the red-hot charcoal. The openings are again closed for a longer period, and when the oven gets a little cooler, more water is added. If any red sparks are observed, the openings and pipes must be carefully stopped up, so as to prevent the formation of a current of air, as this would occasion the combustion of the charcoal and consequently lessen the product.

Great care has to be exercised in accurately regulating the access of air, since the smallest quantity of atmospheric oxygen which passes through the fire-room without being consumed causes a corresponding loss of material in the space where the

FIG. 57.

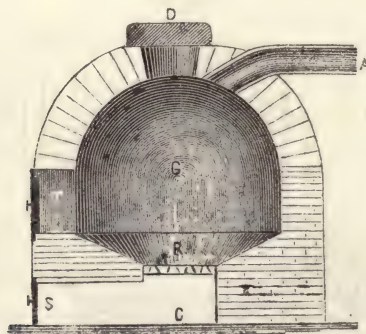


wood is carbonized. Since notwithstanding the utmost care and attention, the access of oxygen can never be entirely avoided, this oven will seldom be used where the chief object is to obtain as large a quantity of acetic acid as possible. An essential improvement in the oven might be made by introducing generator gases in the heating places and burning them by the admission of just a sufficient quantity of air, which could readily be accurately regulated, so that a slight reducing atmosphere would always prevail in the oven.

*Reichenbach's oven*, Fig. 57, is a square construction with double walls, the inner wall of fire-brick and the outer of ordinary brick. The space between the two walls is filled

with sand. The oven is heated by pipes about 2 feet in diameter which run from one end of the wall to the other, and are seen in the illustration at *a*, *b*, *c*, *d* and *m*, *n*, *o*, *p*. The apparatus having been filled with wood, the upper portion is covered with a layer of sods and earth, or with iron plates, the joints of the latter being carefully luted with clay. A fire is then lighted in the fire-places in front of *p* and *d*, which raises the temperature of the pipes so high as to cause them to glow. The wood in the surrounding spaces of the oven abstracts the heat and is thereby carbonized, the volatile products of which pass off at the bottom of the oven through the openings at *x*,

FIG. 58.



into the conduit *f*, *g*, *h*, and through *y* at the opposite side into a similar conduit. Both products intermix in the pipe *ki*, where the tar is partly deposited. From the pipe *ki*, the acetic acid vapors are carried off to the condenser.

*Swedish oven.* This oven is shown in Fig. 58. The space *G* where the wood is carbonized is vaulted and is provided on top with an aperture for charging the wood, which after the vault is filled, is closed by a heavy lid luted with clay. The pipe *A* serves for carrying off the products of distillation. The bottom of the vault is conical, and in the center is provided with the grate *R*, below which is the ash-pit *C*, which can be closed by an accurately-fitting slide *S*. The door *T*, which is bricked

up during the process of carbonization, serves for withdrawing the finished charcoal, and also for the introduction of a portion of the wood. It is immediately closed after the introduction through it of some glowing coals. The effect of the latter is to ignite a portion of the wood, and combustion is conducted by setting the slide *S* so that the vapors are cooled with a certain uniformity. In a short time the wood and the walls of the oven become so thoroughly heated that combustion can be entirely interrupted by closing the slide *S*, distillation being completed by the heat accumulated in the oven.

An oven of a somewhat different construction is shown in cross-section in Fig. 59, and in ground plan in Fig. 60. The

FIG. 59.

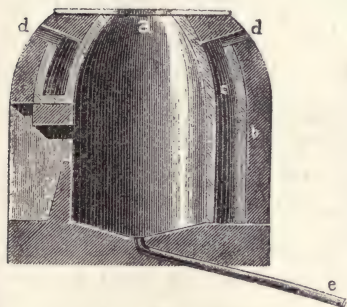
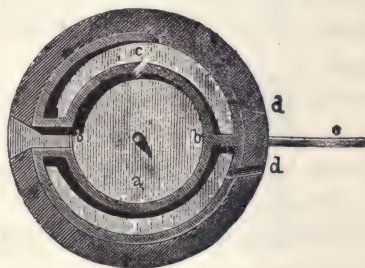


FIG. 60.



space in which the wood is carbonized is in the form of a cylinder and passes above into a vault closed by a heavy iron lid *a*. The brick work of this space is surrounded by another brick work *b*, and the fire which is ignited at the opposite side *d* circulates between the two walls in the space *c c*. On the upper portion of the oven, at *d*, are apertures provided with slides, which serve for regulating the fire. The products of distillation escape through the pipe *e* on the bottom of the carbonizing space.

The oven is heated so that the interior wall becomes red-hot. Firing is interrupted when no more vapors escape from the pipe *e*. The slide at *d* is then closed and the oven left to cool



until the charcoal is sufficiently cooled off to allow of it being withdrawn without fear of ignition.

*Carbo-oven.*—In this oven carbonization is effected in a wrought-iron vertical cylinder with a capacity of 300 to 400 cubic meters of wood. The wood is introduced through openings in the wrought-iron cover of the cylinder. The products of distillation pass out through a pipe branching off from the lowest part of the bottom. An outlet on the side of the cylinder serves for emptying the latter.

Heating is effected by fire-gases produced in a separate furnace, which pass through spiral flues surrounding the wrought-iron cylinder. The lower portion of the cylinder is protected by brickwork from the direct action of the fire-gases.

In the center of the cylinder stands a large vertical heating-pipe divided into halves by a partition. The non-condensable gases, as well as the air required for their combustion, can pass in through two pipes entering the lower part. The smoke-gases coming from the last flue may also be conducted through this heating pipe and, mixed with the combustion products of the wood-gases, escape to the chimney.

*Retorts.*—The various forms of apparatus for the destructive distillation of wood previously described are of such a construction as to exclude uninterrupted operation. When a charge has been distilled off, the kiln or oven has to stand till the charcoal is sufficiently cooled off to allow of its being withdrawn. When this has been done the oven must be again charged, heated and so on. This is evidently connected not only with considerable loss of time, but also of heat, and it has been endeavored to overcome this drawback by the use of retort-ovens.

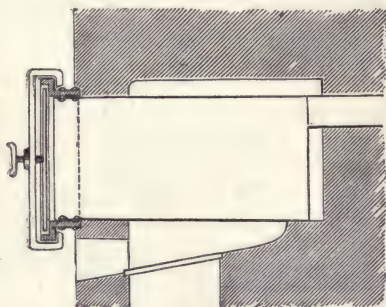
By heating wood in a retort closed air-tight with the exception of an opening for the escape of the products of distillation, and by fitting to this opening a condenser of suitable construction, an apparatus is obtained with which all vaporiform products escaping from the wood can be recovered. Such an apparatus, though the most expensive, is the best for the production of wood vinegar.

a. *Horizontal retorts.*—The arrangement of an apparatus for the distillation of wood is very similar to that used for the production of illuminating gas from coal, the essential difference being that the retorts for the distillation of wood must lie in fire-places which allow of the heat being slowly and uniformly raised, while in making illuminating gas rapid raising of the heat is required. Clay being a worse conductor of heat than iron, the use of retorts of this material would apparently seem advisable for the destructive distillation of wood. However, clay retorts have the drawback of being fragile and besides cracks are readily formed through which a

FIG. 61.



FIG. 62.



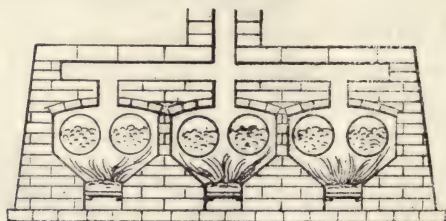
portion of the vapors would escape. For this reason iron retorts are as a rule used. Cast iron retorts do not readily burn through and are but little affected by the vapors of the acid, but they have the drawback of great weight, and defective places are difficult to repair. The best material for horizontal as well as vertical retorts is hot-riveted boiler plate about 8 millimeters thick. Defects in such a retort can be readily repaired by riveting a piece of boiler plate upon the defective place.

A wrought-iron retort of very suitable construction is shown in Figs. 61 and 62. It consists of a cylinder 2.2 meters long and 1 meter in diameter. On the back end the retort passes,

as seen in the illustration, into a pipe of such a length that about 30 centimeters of it project from the brick work of the oven. In front the retort is secured to a cast-iron ring, in the groove of which fits a sheet-iron door. This door is pressed against the ring by a screw and the joint is made air-tight by luting with clay. For the rapid withdrawal of the charcoal, the interior of the retort is furnished with a sheet-iron disk supported by two rods riveted to it. To the center of this disk a chain is fastened which lies upon the bottom of the retort. When the door of the retort is opened the chain is seized with a hook and on being drawn forward the sheet-iron disk pushes the charcoal out.

To protect the portion of the retort which comes in direct contact with the flame it is advisable to apply to it repeatedly

FIG. 63.



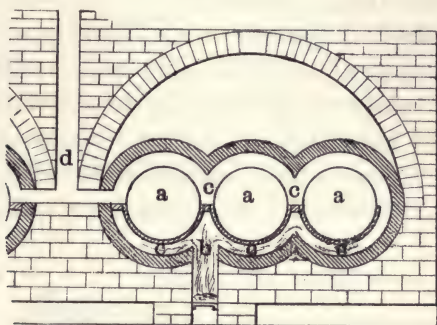
a mixture of clay and cow hair. This prevents quite well the formation of burnt iron which scales off from the portions directly heated.

Fig. 63 shows the manner of bricking in six retorts, two being placed in one fire place. In this construction, as will be seen from the illustration, the fire-gases pass directly into the chimney which is equivalent to a waste of heat. However, this heat can be completely utilized either by placing upon the retort-oven a pan in which the solution of crude sodium acetate may be evaporated, or the fire-gases may be used for heating a room in which the wood for the next operation is dried. In working wood very poor in water, a smaller



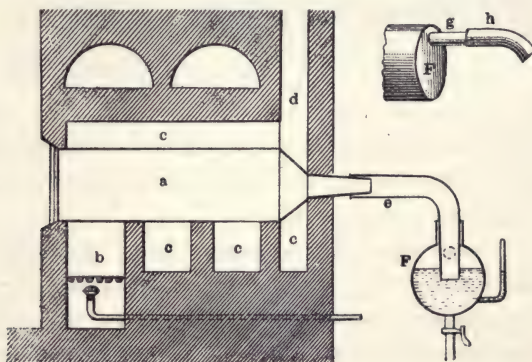
quantity of wood vinegar is to be sure obtained than with the use of ordinary air-dry wood, but it is correspondingly richer in acetic acid.

FIG. 64.



In Figs. 64 and 65, *a a a* are the wrought-iron retorts, *b* the hearth, *c c* the flues, *d* the chimney. Over the somewhat conical neck of the retort is pushed an elbow pipe *e* which

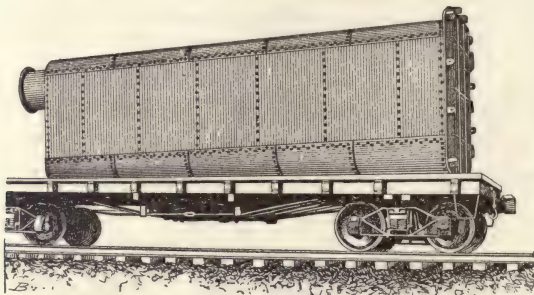
FIG. 65.



dips into the receiver *F*. The latter is a cast-iron pipe 1 to 2 feet in diameter according to the number of retorts, and extends the entire length of the oven. For the neck of each retort it carries a tubulure  $5\frac{3}{4}$  to  $7\frac{3}{4}$  inches long. The object

of the receiver is to receive the products of distillation from all the retorts and at the same time to hydraulically close the elbow-pipe of each receiver. Hence the vapors not precipitated in the receiver can continue their way through *g* to the other condensing apparatus *h*, but cannot re-enter the retorts. This is of no slight importance, for if there were no water-joint and the vapors should from any cause suddenly cool off, the external air might penetrate into the retort, and the latter being filled with inflammable gases and vapors of a high temperature, an explosion would necessarily follow. For making the water-joint it suffices for the elbow-pipes to dip  $\frac{3}{4}$  to 1 inch

FIG. 66.



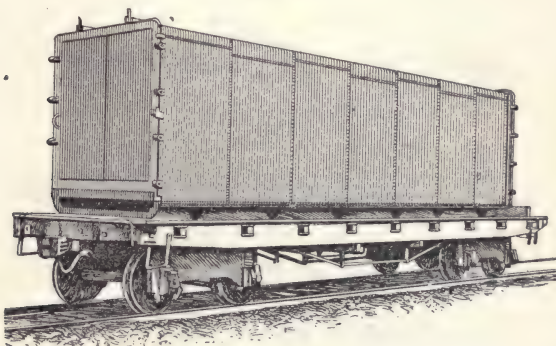
into the fluid into the receiver. But as the fluid constantly increases, provision must be made for its discharge through a pipe, placed below or on the side, into a collecting vessel located in another apartment.

In many plants the gases escaping from the condenser are utilized for heating by conducting them under the retorts through a suitable pipe system. However, the pipe-system should be so arranged as to allow of the gas being conducted under any one of the retorts or being shut off from it in case of necessity, because if distillation progresses too rapidly, the fire under a retort may have to be entirely removed in order to moderate the chemical process in the retort. It is advisable to arrange the gas conduit so that the gas can also be used for heating other apparatus, for instance, evaporating pans, etc.

In this country what is known as the oven-retort, Fig. 66, is largely used in equipping plants for hard-wood distillation. This retort is a rectangular wrought-iron chamber, a common size being 6 feet wide, 7 feet high and from 27 to 50 feet long, according as it is intended for two or more cars loaded with wood. The oven is set in brickwork or is made with double iron walls with an air space between. It is provided with a large door closing air-tight, and is heated by wood, charcoal, coal or gas.

When distillation is finished the cover of the retort is removed, and the glowing charcoal is with the assistance of the

FIG. 67.



previously-described contrivance emptied into cans, which are immediately closed with tightly-fitting covers, the latter being luted with clay or sand to prevent ignition of the glowing coal by the entrance of air. If such retorts as shown in Fig. 66 are used, coolers, Fig. 67, similar in shape are employed, in which the coal is allowed to remain until thoroughly cool.

b. *Vertical Retorts.*—The principal drawback of horizontal retorts is that, on the one hand, charging them is connected with some difficulty in case they are longer than two lengths of the wood, and, on the other, that defects in them cannot as a rule be immediately detected, and that when repairs have to be made they have to be taken from the oven. In addi-

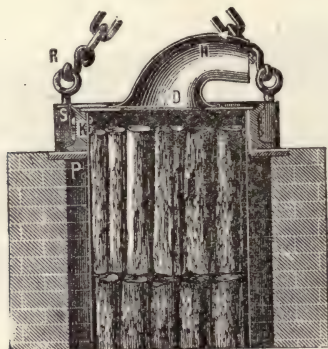


tion a certain number of sheet-iron cans have to be provided for the reception of the charcoal drawn from the retorts.

The arrangement of the retorts so that they can be lifted from the oven and replaced by others has many advantages. The operation can be carried on without interruption by removing a retort in which distillation is finished and replacing it by another, in which distillation at once recommences, because the hot brickwork throws out heat continuously, and heating need to be interrupted only during the time required for lifting out the retort and replacing it by another.

The accompanying illustrations show the arrangement of the

FIG. 68.

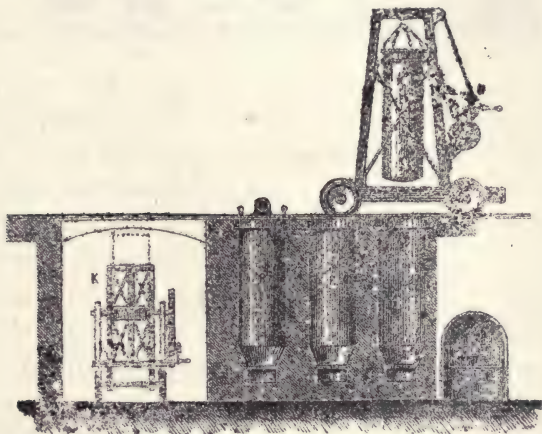


retort-ovens and the lifting apparatus as devised by Dr. Josef Bersch. Fig. 68 shows a retort 12 feet high with a diameter of 3 feet and 3 inches. It is constructed of boiler-plate 0.315 inch thick, the bottom, *i. e.*, the portion which is exposed directly to the fire, being of plate 0.394 inch thick. The upper portion of the retort is provided with a cast-iron ring *K* which, when the retort is lowered into the oven, rests upon a flat cast-iron ring *P* placed upon the brickwork. On this ring are four eyes *S* which serve for fastening the lifting tackle, and the lid of the retort is secured by four pins pushed through the openings in the ring. The lid of the retort consists of a sheet-iron disk, provided in the center with a conical head-

piece *D* which terminates in the pipe *H* leading to the condenser.

Figs. 69 and 70 show the retorts placed in the oven and the mechanical contrivance for raising and lowering the retort *R*. On top of the ovens is a track *C*, upon which runs a crane with a head-piece having the form of a truncated cone. The track is continued from the last oven to a brick wall upon which it rests, and beneath this track is another one, which runs to the place where the charcoal is to be emptied and the retort is to be re-filled with wood. The function of this mechanical contrivance

Fig. 69.

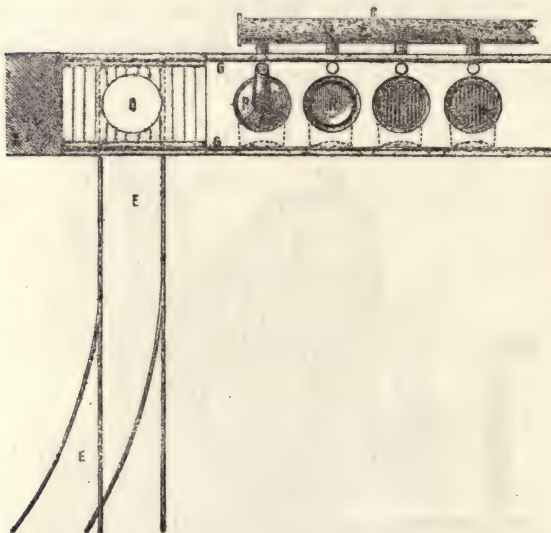


is as follows : The retort, after the contents have been distilled, is, while hot, lifted from the oven by pushing the crane over it and drawing it into the hollow pyramid. The crane is then pushed over the opening *O*, upon which stands a carriage *K* upon the other track *E*. The carriage is provided with a basket-like arrangement for the reception of the retort. The retort having been lowered into the basket, the latter is brought into a horizontal position by turning a screw without end. The carriage, which is actually a dumping-car, is pushed over the pit for the reception of the charcoal, and the retort, the lid of which is now taken off, is sufficiently inclined to allow the char-

coal to fall into the pit. The charcoal is protected from ignition by being covered with wet charcoal dust. The empty retort is then again brought into a horizontal position and refilled with wood.

While the retort just coming from the oven is thus handled, and the first dumping-car has been pushed away, another dumping-car is immediately brought from a side track *E* under

Fig. 70.



the crane, the retort lifted in, the crane pushed over the empty oven, and the retort lowered.

Since towards the end of distillation the greatest heat must be applied in order to obtain the last remnants of acetic acid and tar, the sides of the oven are hottest at this period. If now immediately after a retort with charcoal has been taken out, another one charged with wood is brought into the oven, the heat radiating from the sides of the oven suffices to induce distillation, and the fire need only be slightly stirred to uninterruptedly carry on the operation.

One crane and two dumping-cars are sufficient for attend-



ing twelve to eighteen ovens arranged one alongside the other. For a larger number of retorts it is advisable to have two cranes, and to arrange the course of the operation as follows: One crane, in the pyramid of which is suspended a retort filled with wood, which has been lifted from a dumping-car standing, for instance, on the right-hand end of the series of ovens, is immediately pushed over the oven as soon as the retort filled with charcoal has been lifted out, and the retort is then lowered. The other crane is pushed to the left-hand end of the series of ovens, where the retort is lowered, and so on.

By the employment of these contrivances the time required for distillation is reduced to a minimum, the operation can be carried on without interruption, and it is not necessary to provide sheet-iron cylinders for cooling the charcoal, since the latter is dumped from the retorts directly into pits between the rails upon which the dumping cars run, where it is cooled by covering with wet charcoal dust.

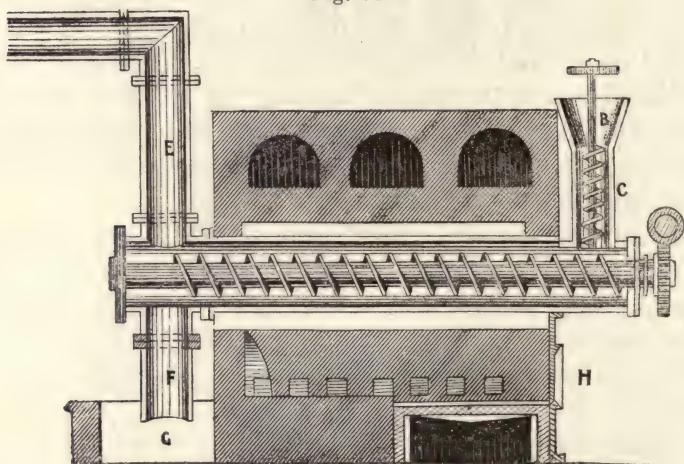
*Distilling apparatus for wood waste.*—In working wood and bark in the various trades a large quantity of waste results which, in most cases, is used as fuel. Such waste can, however, be utilized to greater advantage by subjecting it to destructive distillation for the purpose of obtaining wood-vinegar, tar and charcoal.

*Halliday's apparatus* for the production of acetic acid, etc., from sawdust, spent bark from tan-yards and dye woods exhausted of their coloring matter, is shown in Fig. 71. The waste to be treated is introduced into a hopper *B* placed above the front end of an ordinary cylinder *C*, in which a vertical screw or worm revolves, conveying the material, and in proper quantities, to the cylinder, placed in a horizontal position, and heated by means of a furnace *H*. Another revolving screw or worm *D* keeps the material introduced into the retort by *C* in constant agitation, and at the same time moves it forward to the end. During its progress through the retort the materials are completely carbonized and all the volatile

products disengaged. Two pipes branch off from the ulterior part of the retort, one *F* passing downward and dipping into an air-tight vessel of cast-iron, or a cistern of water *G*, into which the carbonized substance falls. The other ascending pipe *E* carries off the volatile products of the distillation into the condenser, consisting of pipes of copper or iron immersed in or surrounded by water.

The arrangement of the cylinder *A* with the screw is similar to the worms used for moving grain, malt, etc., in a horizontal direction. According as the screw revolves with

Fig. 71

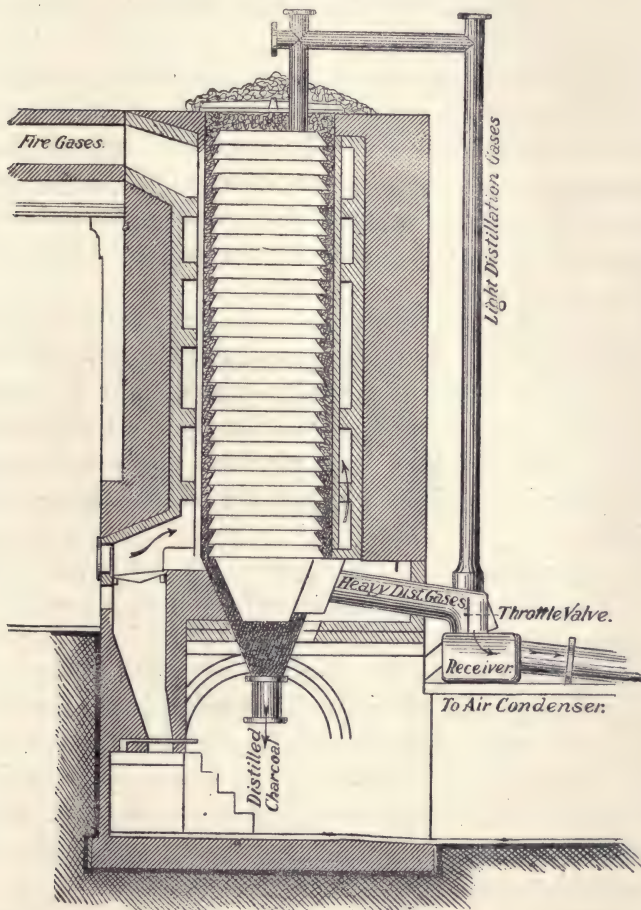


greater or less rapidity, the materials can for a shorter or longer time be exposed to the action of the heat, and according to well authenticated statements, the quantity of acetic acid obtained from the wood substance distilled in this apparatus is larger than that derived from blocks of wood stacked in other retorts.

This fact, however, cannot be ascribed to the construction of the apparatus, which is not particularly favorable, but is exclusively due to the condition of the wood. From the small particles of wood the products of distillation escape with far

greater rapidity than from the large blocks, which must be very hot on the surface before their interior is sufficiently heated for distillation to commence. Hence the products of

FIG. 72.



distillation must pass through the strongly-heated carbonized parts, whereby a considerable portion of the acetic acid is decomposed.

Another apparatus suitable for the distillation of sawdust,



spent tan-bark, exhausted dye woods and waste of wood in general, is shown in Fig. 72. It consists of an iron cylinder, 18 feet high and  $5\frac{1}{2}$  feet in diameter, which contains a number of bell-shaped rings placed one above the other. In this manner a kind of annular cylinder is formed which below terminates in a conical space.

The materials thrown in at the top are heated in the cylinder, and the vapors in the cavities of the bell-shaped rings pass upwards, while the charcoal falls down and is from time to time removed. In removing the charcoal, the lower portion of the cylinder is closed by a slide, so that by introducing material on top of the cylinder, distillation can be carried on without interruption.

The small charcoal resulting in the destructive distillation of wood waste, may be utilized in various ways. A portion burned upon a grate of suitable construction, for instance, a step-grate, serves as fuel in the factory itself, while the remainder, especially that from sawdust, forms in the finely divided state in which it is turned out, an excellent disinfecting agent.

The various apparatus employed in the destructive distillation of wood having now been described, it may be stated that it is impossible to say which is to be preferred, this depending largely on local conditions. The decision must particularly be influenced by the fact whether the charcoal is of value or not. In the first case it will evidently be of advantage to employ smaller apparatus, so arranged that besides thoroughly carbonized charcoal, all the wood-vinegar and tar are obtained, and further, that the resulting gases can be employed for heating the retorts.

But where the conditions are such as to make it difficult to realize on the charcoal, the principal profit of the plant will be in the yield of acetic acid and wood spirit, and for this reason it is best to carry on the destructive distillation of wood in very large retorts, since with their use the temperature can be raised very slowly, whereby wood-vinegar very rich in acetic acid is obtained.

*Coolers.*—The products which escape in the destructive distillation of wood consist, in addition to acetic acid, water and other very volatile products, of very large quantities of gas. Since the current of gas is the carrier of vapors, and considerable quantities of gas are evolved at certain stages of the process, provision must be made for the thorough condensation of the vapors to prevent the escape into the air of large quantities of valuable products of distillation, or their being burned with the gases.

In plants working with a number of retorts, the discharge pipes of the latter enter into a common pipe of large diameter, and in this condensing pipe, which in a short time after the commencement of the operation becomes very hot, the vapors and gases entering it are cooled off to a considerable extent, since the condenser in consequence of its large surface yields considerable heat to the surrounding air. A portion of the heavier volatile tar products is already condensed in the condenser, and is drawn off by means of a faucet in the lowest portion of the pipe. It might be suitable to place over the condenser a pipe with numerous small perforations, so that a spray of water in such quantity that it immediately evaporates, falls constantly upon the condenser.

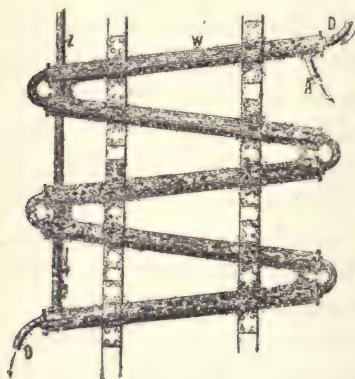
By the use of this contrivance not only a large portion of the vapors are liquefied, but another advantage is attained. Since by this cooling the tension of the vapors and gases in the condenser is diminished, the vapors formed in the retorts pass out with great rapidity. This is of great advantage, since by the vapors remaining for a long time in the retort a considerable quantity of acetic acid is decomposed. Furthermore, the volume of non-condensed vapors is considerably decreased, so that a cooler of smaller dimensions can be used, than would be possible if all cooling had to be done in it.

*Counter-current Pipe Cooler.*—The arrangement of such a cooler is shown in Fig. 73. The pipe *D*, containing the vapors to be cooled, is surrounded by another pipe *W*, filled with water. From the reservoir placed at a higher level, cold water

is conducted through the pipe *Z* to the lowest part of *W*, and passing through the latter, runs off at *A*. Since the vapors in the lowest part of *D* have already been cooled off to a great extent, they yield but little heat to the water. As the water reaches the higher portions of *W*, it constantly acquires a higher temperature from the heat withdrawn from the vapors, and finally runs off at *A*. With a sufficient length of the cooling pipes and a powerful current of water, the vapors are so completely condensed that but very small quantities of acetic acid and wood spirit are carried away by the current of gas.

To obtain this acetic acid, the gas before being burned is

FIG. 73.



allowed to pass through a cylinder *d*, see Fig. 76, which is from  $3\frac{1}{2}$  to 4 feet high and filled with limestone. The acetic acid contained in the gas is fixed by the limestone and the calcium acetate thus formed can be obtained by lixiviation.

The cooling pipes should be of considerable length. With six retorts in operation at the same time, the length of the pipes should be about 130 feet and their diameter  $5\frac{3}{4}$  inches, since otherwise great pressure is caused in the apparatus by back pressure of the gases, which results in a decrease in the yield of acetic acid. Hence it is advisable to arrange the upper portion of the cooling apparatus so that the pipe *W* has



an elliptical cross section and contains two pipes *D*, one alongside the other, which in passing out from this portion combine to one pipe.

To prevent obstruction in the pipes by the collection of viscous tarry substances, it is recommended to give them considerable inclination and to connect them so that, in case of necessity, the interior of each pipe can be cleansed with a brush. Fig. 74 shows the most suitable way of connecting two pipes. The upper pipe is connected with the lower one by means of a curved joint secured by screws.

To prevent the fluid running off from the cooler from being

Fig. 74

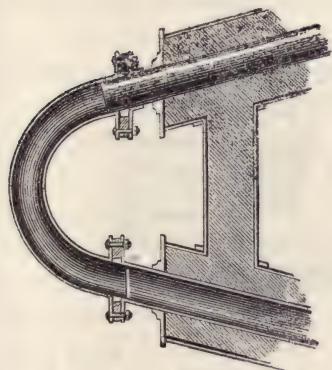
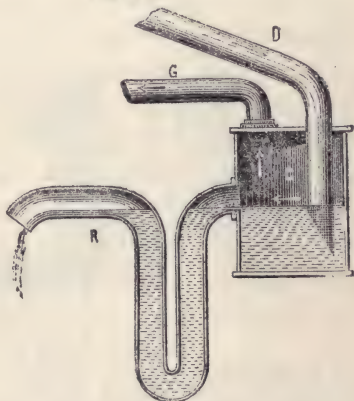


Fig. 75

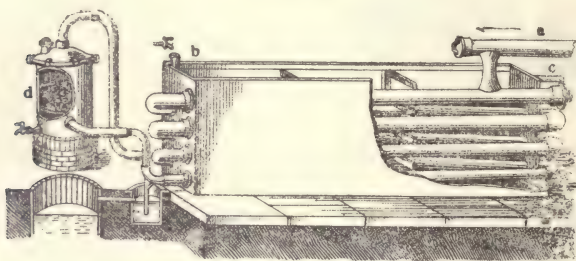


forced by fits and starts from the lower pipe by the current of gas, the contrivance shown in Fig. 75 may be used. The pipe *D*, coming from the cooling apparatus, is cut off at an acute angle, and extends nearly to the bottom of a cylindrical vessel *C*, to which is fixed a U-pipe *R* at such a height that the fluid in *C* can rise to the upper edge of the cut end of *D*. The pipe *G*, fixed in the lid of the cylinder, carries away the gas from *C*. Since with an increase in the development of gas, the latter, in order to escape, needs only to press down a layer of liquid of very moderate height, it can pass off without impediment, while the distillate which collects in *C* runs off through *R*.

If neither liquid nor gas escapes through *D*, the discharge of wood-vinegar from *R* ceases at once, and the mouth of *D* is closed.

*Box-cooler*.—In place of the counter-current cooler, the box-cooler shown in Fig. 76 is used in some establishments. In a long, narrow trough or box of wrought-iron or wood lies a series of straight, wide, copper pipes with a gradually decreasing diameter. The pipes are slightly inclined, so that the fluid, running in at the highest point, flows out at the lowest. Outside the trough the pipes are connected by movable elbow joints. One end of each pipe is firmly fixed to the wall of the trough, while the other, to prevent free expansion, sits loosely

FIG. 76.



in a slightly conical socket. The lower end of the last pipe divides into two branches, one of them leading downward and dipping into the receiver, while the other, as a rule, conducts the gases directly under the fireplace. There should be but a small space between the collecting pipe *a*, which conducts the vapors to the condenser, and the first condensing pipe, as otherwise obstructions might readily be formed by the deposit of tar dried by the hot vapors. A constant stream of water is conducted through *b*, along the bottom of the trough, the heated water running off at *c*.

The development of gas from the wood being very irregular and by no means in the proportion desirable for the heating of the retorts, it is preferable to collect it in a gasometer and

distribute it from there as may be necessary, instead of conducting it directly into the fire. But little gas is developed in the beginning of the operation, and much towards the end, while the reverse proportion is desirable.

In case condensation is not very complete, the pipe leading to the hearth or gasometer is more or less attacked by acetic acid precipitated in it by the access of air. To prevent this evil it is advisable to place on the pipe small receptacles provided with cocks for the collection and discharge of any fluid deposited. These receptacles may also be filled with quick lime, which at least fixes the acetic acid, thus rendering it harmless for the pipe. The lime is from time to time extracted with water to regain the soluble calcium acetate.

To further cool off the current of gas and render the vapors of acetic acid carried along with it harmless for the pipe, Vincent uses a cylindrical copper receptacle, *d*, Fig. 76, provided with a false bottom, upon which is placed a layer of crystallized soda from  $2\frac{1}{4}$  to  $2\frac{1}{2}$  feet deep. The vapors of water and acetic acid dissolve the soda, and the temperature thereby being lowered, a further portion of the volatile bodies, especially wood spirit, is precipitated. By distilling the fluid thus obtained, the wood spirit is regained, and the residue in the still used for the preparation of sodium acetate.

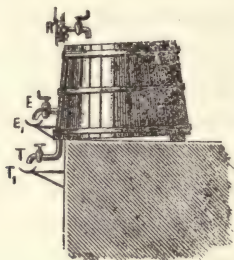
For a condenser for four retorts of a capacity of 141.26 cubic feet each, Gillot gives the following approved dimensions, provided the period of distillation is 72 hours: The diameter of the pipe at its entrance into the water trough is  $15\frac{3}{4}$  inches, and at its exit  $5\frac{3}{4}$  inches; its total length is 164 to 180 feet, this length being divided between 6 straight pieces and their elbow-joints. The vat is  $26\frac{1}{4}$  feet long with a depth of  $5\frac{1}{4}$  feet.

*Reservoirs for the Product of Distillation.*—For storing the liquid products of the destructive distillation of wood, wooden vats 6 or more feet high with a capacity depending on the quantity of the daily distillate are generally used, it being advisable to have them of such a capacity that at least one of



them is filled every day. The U-pipe through which the fluids run off from the cooling apparatus terminates over a funnel fixed to a pipe which runs alongside the vats, and is provided

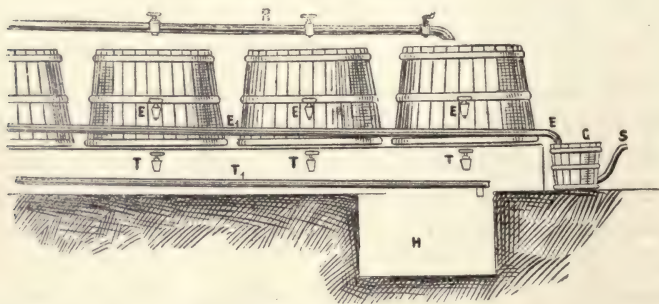
FIG. 77.



on the corresponding places with stop-cocks by means of which the fluid can be discharged into any vat desired.

Each vat is placed so as to incline slightly forward, and on the lowest place is provided with a cock, beneath which is a gutter. About 8 to 10 inches above the bottom of each vat is a cock with a gutter underneath. Figs. 77 and 78 show the arrangement of the vats. The pipe *R* serves for filling the

FIG. 78.



vats with the products of distillation; the cocks *T* and the gutter *T1* for discharging the tar into the brick reservoir *H* sunk in the floor; the cocks *E* and the gutter *E1* for drawing

off the wood vinegar into the small vessel *G*, to which is secured the suction pipe *S* of a pump for the further conveyance of the wood vinegar.

It is advisable to coat the vats and iron hoops with hot wood tar.

*Collecting boxes.*—When working on a large scale quite a number of vats are required, which involves considerable expense, together with the disadvantage of occupying considerable space. It is therefore advisable to use in place of vats collecting boxes sunk in the ground.

Such boxes are best and cheapest constructed of about 3-inch wooden planks, every kind of wood being suitable for the purpose, since the products of distillation with which the boxes become saturated preserves them even in moist soil. From the planks prismatic boxes, each about 13 feet long, 13 feet wide and 8 feet deep are constructed. The boxes are sunk in the ground, and in the corner of each box is placed a small barrel *F*, Figs. 79 and 80, into which can be dipped the suction pipe of a pump. The spaces between the planks and the walls of the pit are filled with earth, and the joints between the planks with pitch. On top each box is provided with a frame, upon which is placed a lid made of planks.

The requisite number of boxes are placed alongside each other so that about 3 feet of ground remain standing between the sides of every two boxes. The products of distillation running off from the cooling apparatus are conducted through a pipe running the length of the boxes into the vessel to be filled. To prevent a box from being filled too full, all the boxes are connected by the wooden pipe *R*, placed about 15 inches below the edge.

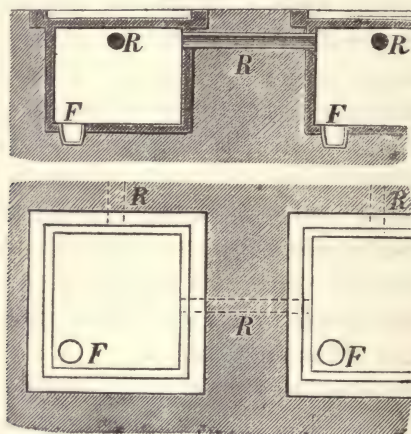
When a box has been several times filled with the products of distillation, the layer of tar deposited on the bottom is of sufficient depth to be pumped out. The suction pipe of the pump is then lowered to the bottom of the small barrel in the corner of the box, and the tar, with the exception of a small portion, can be separated from the wood vinegar by pumping.

Figs. 79 and 80 show the arrangement of several such collecting boxes in ground plan and elevation.

Since the separation of the tar from the wood vinegar takes place the more completely the longer the fluids are allowed to repose, it is advisable to first fill all the boxes in turn with products of distillation and then to work further the contents of the box filled first.

*Utilization of the Gases.*—The gases evolved in the destructive distillation of wood may advantageously be used as fuel. In the commencement of the operation a gas mixture, very rich

FIGS. 79-80.



in carbonic acid, is obtained, which is of little value as fuel, but latter on less carbonic acid is evolved and the gas contains, besides carbonic oxide, hydrogen and hydrocarbons, which are of considerable value as fuel.

The most suitable plan would be to catch the gases by means of a pump from the pipe *G*, Fig. 75, and collect them in a gas holder of ordinary construction, and to conduct them from the latter by means of pipes to the fire-place. However, a gasholder of sufficient capacity to hold the large quantities of gas evolved would be rather an expensive affair for a plant engaged in the distillation of wood, and it is therefore gener-



ally preferred to conduct the gas directly to the fire-places where it is to be burnt.

When working with a large number of retorts, the operation may be so conducted that the wood or coal fire under the retort just placed in the oven is allowed to go out entirely, and to fire only with gases escaping from retorts in which distillation is in full progress, and from which a large quantity of gas is constantly evolved. In factories devoted to the further working of the wood vinegar, it is best to conduct the gases of distillation under an apparatus which has to be heated almost without interruption, for instance, under the pans in which the crude sodium acetate is evaporated to crystalization.

Care should be taken not to ignite the gas escaping from the retorts before all the air has been displaced from the entire apparatus—retorts, condenser and cooler—since otherwise an explosion might take place by the flame spreading into the pipe conduit, which would not only be dangerous, but sufficiently heavy to tear apart the lute of clay on the pipes for the vapors, the condensers, etc. When the vapors escaping from a retort condense on cooling to a yellowish colored fluid, all the air has been displaced from the apparatus, and the gas may be ignited without fear of danger.

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## CHAPTER XXIII.

### EXECUTION OF THE DESTRUCTIVE DISTILLATION OF WOOD.

No matter what the arrangement of the apparatus may be in which the destructive distillation of wood is to be carried on, the course of distillation as regards the succession of phenomena remains the same, and a distinction has only to be made in reference to the kind of product desired, and the duration of time for the operation. The latter depends on the quantity of wood used at one time ; the larger the latter is, the

longer the operation will have to be continued, and under otherwise equal conditions, more time will be required for the complete distillation of a charge of wood, if wood vinegar, tar and black charcoal are to be obtained.

When working with a larger number of retorts, the operation should be so arranged that it is carried on uninterruptedly, this being advisable on account of the division of labor, and also to prevent being forced to adopt special expedients by reason of the vast quantity of gas evolved at a certain stage of the operation.

With the use of vertical retorts and a suitable lifting tackle, the retorts are placed open in the oven and a gentle fire is started. At first only steam evolves from the wood, which is allowed to escape into the air, and only when a peculiar aromatic odor indicates the commencement of distillation, are the lids placed upon the retorts and connected with the cooling apparatus. To make the lid steam-tight, a roll of clay is laid upon the edge of the retort and the lid pressed down upon it, the clay forced out thereby being smoothed down with an elastic steel blade.

The time during which distillation has to be continued depends on the size of the retorts, but, as a rule, the operation is so conducted that distillation is finished in 12 hours. Of course, with the use of very large apparatus in which a great quantity of wood is carbonized at one time, distillation requires several days, since the heating of such a large quantity of wood to the temperature of decomposition takes considerable time. When the temperature—about 393° F.—has been reached at which the wood begins to yield more abundant quantities of products of distillation, care must be taken to keep the fire under the retorts so that the temperature increases gradually and reaches 662° F. only in the last period of distillation, so that with a distilling time of 12 hours, the temperature in the retorts remains for about ten hours below 662° F. To gain practical experience in regulating the temperature in heating, which is of special importance with new

ovens, it is advisable to place a thermometer on one of the retorts as follows: In a small aperture in the lid of the retort is screwed an iron pipe closed below and open at the top. The length of the pipe should be such that the lower end reaches to the center of the retort. In this pipe is lowered by means of a wire a thermometer graduated to 680° F. (the boiling point of mercury). By from time to time consulting this thermometer, a conclusion can be drawn as to the degree of heat prevailing in the retort. Experienced men can accurately judge of the progress of distillation from the quantity of distillate running off, and of the gases escaping simultaneously.

When, for instance, 5 cubic meters of wood—the contents of two retorts—are to be distilled at one time, the first distillate, in a jet about the thickness of a lead pencil, is obtained with correct firing, in about 1½ to 2 hours after the commencement of heating. The thickness of the jet of fluid does not change for hours, and it retains its original yellow color. The gas issues in a moderately strong current from the respective pipe and burns with a pale blue flame, the latter becoming more luminous only later on at a higher temperature when hydrocarbons are mixed with the gas.

When a temperature of about 662° F. has been reached, the quantity of distillate suddenly becomes smaller, and the quantity of escaping gas also decreases. In order to obtain the last remnants of the product of distillation, which consist predominantly of tar products, the fire is increased, when a more abundant quantity of distillate is obtained. However, the jet of fluid running off from the cooler is henceforth of nearly a black color, due to numerous drops of dark-colored tar products. The volume of gas becomes larger and these gases burn with a very luminous, pure white flame. In this last stage of distillation certain precautions have to be observed in reference to raising the temperature. If it is raised too rapidly at once, such a large quantity of gas is evolved from the retorts as to cause a high pressure in the apparatus, which is recognized by the force with which the current of gas issues



from the pipe entering the fire-place. Since in this stage of the process the retorts are hottest and their bottoms not seldom red hot, there is danger of the riveted places becoming leaky, so that in the succeeding operations a considerable quantity of products of distillation is lost by its escape in the form of vapor through these leaky places and being burned.

When the volume of gas is observed to become greater, about  $1\frac{1}{2}$  hours before the end of distillation, the fire under the retort may be allowed to go out entirely, since the heat developed in the retorts in consequence of decomposition in conjunction with that radiating from the sides of the ovens, suffices to finish the operation. When the temperature in the retorts rises to  $806^{\circ}$  F., the evolution of products of distillation ceases almost suddenly and the retorts now contain only black charcoal.

Since antimony melts at  $809.7^{\circ}$  F., this metal may be used for determining the commencement of the end of distillation. For this purpose the thermometer is removed from the pipe previously mentioned, and a small crucible containing a piece of antimony is lowered by means of a wire into the pipe. When the antimony is melted, distillation may be considered finished and the retort be at once lifted from the oven.

The fluid which runs off during distillation is at first wax-yellow, but later on becomes of a darker color, red brown, and finally nearly black, and is quite turbid. When allowed to repose it separates into two—or perhaps more correctly into three—layers, sharply separated one from the other. The lowest layer is tar, a thick fluid of a dark, generally pure black color; the middle layer, which comprises the greater quantity, is wood vinegar, and is of a red yellow or red brown color. The upper layer is again of a dark color, and possesses the properties of tar, but, as a rule, this tarry mass is present in such small quantities that it even does not cover the entire surface, but swims upon it like flakes.

It is advisable to allow the distillates to repose for a considerable time, the tar thereby separating more completely from

the wood vinegar, and the latter is obtained as an entirely clear red brown fluid, which can be manufactured into acetic acid with greater facility than wood vinegar mixed with larger quantities of tar.

By giving the vats intended for the reception of the distillate such a size that one vat is filled by the distillate obtained in one day, and arranging twelve such vats as shown in Fig. 78 the contents of the vat filled first can be allowed to repose for 11 days before the manufacturer is forced to empty it in order to make room for fresh distillate. When the wood vinegar and tar are to be worked further in the factory itself, the apparatuses intended for this purpose should be of such dimensions that the quantity of wood vinegar produced daily can be worked up at one time. The quantity of tar being considerably smaller than that of wood vinegar, it is collected in the reservoir *H*, Fig. 78, and larger quantities of it are worked in one operation.

When the liquid products of distillation are caught in boxes sunk in the ground, described on p. 281, the operation may be so arranged that the distillate is allowed to run uninterruptedly into the first box until it is filled with tar to such an extent that not only wood vinegar, but also tar commences to run off through the pipe *R*, Figs. 79 and 80. The tar is then allowed to repose for some time, whereby it still better separates from adhering wood vinegar, and the larger quantity of pure tar thus obtained is then worked up at one time.

Experience has shown that it is of advantage to allow the tar also to repose as long as possible, it having been observed that if kept for some time in special reservoirs, a permanent separation of the products according to their specific gravities takes place. On the bottom of the reservoir tar of a very viscous gritty nature collects, which is of such thick consistency that it can scarcely be raised by a pump. The higher layers of the tarry mass are of thinner consistency, the uppermost being almost oleaginous, and upon them floats a layer of

wood vinegar, which can from time to time be taken off and worked together with that drawn from the vats.

*Yield of Products.*—The quantities of wood vinegar and tar which are obtained from a given quantity of wood depends on several factors, namely, on the kind of wood, its content of water, and the manner in which distillation itself has been conducted. Since these three factors vary very much, it is evident that there must be considerable differences in the statements regarding the quantities of products of distillation, and especially the quantities of acetic anhydride and wood alcohol which can be obtained from crude wood vinegar, because by careless manipulation a large quantity of the acetic acid present in wood vinegar is lost.

In order to obtain accurate data regarding the quantities of wood vinegar and tar which can be obtained from a variety of wood when working on a large scale, it is necessary to weigh the wood worked during a certain time, to determine its content of water, to ascertain in a sample of the wood vinegar resulting from each distillation the quantity of acetic acid and wood alcohol, and finally to accurately ascertain the volume of vinegar. From the results of such a series of tests and from the quantities of pure acetic acid and wood alcohol furnished by the factory itself, it would be possible to obtain reliable data regarding the quantities of products of distillation which may be obtained from a given variety of wood.

Stolze has published experiments made with the greatest care to show the amount and strength of the products obtained from the distillation of several kinds of wood. The quantity of each kind of wood submitted to destructive distillation was *one pound*, a quantity suitable, in most cases, to form a precedent for the manufacture on a large scale. The woods were all collected at the same time of the year (towards the end of January) and only those of nearly the same growth were chosen. From Stoltze's table the following figures for the most important varieties of wood have been calculated :



	Wood vinegar, pounds.	Therein acetic anhydride, pounds.		Tar, pounds.	Char- coal, pounds.	Gases, cubic meters.
100 pounds of birch .....	44.9	8.9	also	8.6	24.4	9.8
100 pounds of beech .....	44	8.6	"	9.5	24.6	10.8
100 pounds of hornbeam.	42.5	7.6	"	11.1	23.9	10
100 pounds of oak .....	43	7.7	"	9.1	26.1	10
100 pounds of fir .....	42.3	4.2	"	11.9	26.6	12.5

The results obtained by Assmus in manufacturing on a large scale are as follows :

100 pounds of—	Yield wood vinegar, pounds.	Which yield calcium acetate, pounds.	Or acetic an- hydride, pounds.	Tar, pounds.	Char- coal, pounds.	Crude light oil, pounds.	Crude heavy oil, pounds.
Birch 25 to 40 years old ....	46	5.2	3.9	8	23.5	1 2	4.5
Birch-bark, first extract	22	0.6	0.4	30	18.5	21.6	3.0
Birch-bark, second extract.	20	0.7	0.5	20	22	12	4.7
Oak .....	42	6.0	4.5	8.8	27.5 (?)	0.8	3.3
Fir .....	42	3.2	2.4	10.5	22	1.3	5.7
Pine .....	44.5	3.0	2.3	9.5	22.6	0.6	3.5

According to Roth's experience, the trunk-wood of birch from 60 to 80 years old and grown upon a high dry soil with a limestone sub-soil surpasses the best red beech in the yield of acetic acid. He obtained from 100 pounds of this kind of wood dried at  $140^{\circ}$  to  $158^{\circ}$  F., with heating for 48 hours, at a temperature not exceeding  $750^{\circ}$  F., 40 pounds of wood vinegar of 25 per cent. acetic anhydride, also 2 or 3 per cent. of tar, and 30 per cent. of red charcoal suitable for the manufacture of powder.

Klar\* gives the following yields obtained with retorts. The figures refer to anhydrous wood of 100 per cent., expressed in per cents. by weight.

\* Technologie der Holzverkohlung, 1910.

	Charcoal.		Calcium acetate of 80 per cent.		Crude wood spirit of 100 per cent.		Tar.		Tar oil.	
	Maximum.	Minimum.	Maximum.	Minimum.	Maximum.	Minimum.	Maximum.	Minimum.	Maximum.	Minimum.
European beech .....	33	28	10.5	8	2.5	1.7	6	5	..	..
American maple .....	35	..	8	..	2	..	7	..	..	..
Very resinous fir .....	33	28	2.5	2.3	0.42	0.28	20	..	..	..
European fir .....	36	33	3.6	3	0.8	0.6	12	6	5	0.4
Sawdust from conifera....	33	..	3	..	0.6	..	10	..	..	..
Olive kernels .....	35	..	4	..	1.2	..	4	..	..	..

## CHAPTER XXIV.

### TREATMENT OF THE WOOD VINEGAR.

Wood vinegar in the state it is obtained from wood finds but a limited application. Without further treatment it can in a crude state be used only for impregnating wood or for the preparation of ferric acetate (red liquor), because a portion of the tar products impart to it a penetrating empyreumatic odor rendering its use for other purposes impossible. It is also not possible to free the wood vinegar from these tar products simply by distillation. By repeated rectification highly concentrated acetic acid remaining almost colorless in the air is to be sure finally obtained, but it has always a more or less empyreumatic odor which makes it unavailable for comestible purposes.

The acetic acid can, however, be separated in a perfectly pure state from the wood vinegar, and upon this is based not only the preparation on a large scale of the various acetates, but also, what is perhaps of still greater importance, the production of absolutely pure acetic acid suitable for comestible purposes.

No matter how the wood-vinegar is to be used, it is of great importance to separate it as much as possible from the tar. Freshly prepared wood-vinegar is a turbid red-brown fluid. By allowing it to stand quietly in a tall vessel a quite thick layer of tar separates on the bottom, over which stands the perfectly clear wood-vinegar; a thin, oleaginous layer of light tar products also floats sometimes upon the surface of the vinegar. As in the further treatment of the wood-vinegar the presence of tar causes many disturbances, care must be taken to separate it as much as possible by mechanical means, this being effected by allowing the products of distillation to stand in the vats for several days—the longer the better.

It has been proposed to heat the wood-vinegar by placing a copper coil in the vat, and thus effect a more complete separation of vinegar and tar. But independent of the expense, the result of this treatment is less favorable than that obtained by allowing the products of distillation to stand for a long time, and besides, by heating too much, a portion of the readily volatile wood spirit is evaporated.

There are several ways by which concentrated acetic acid can be obtained from crude wood-vinegar, the one to be selected depending on what the acetic acid is to be used for. When it is to be employed for the production of acetates where a slight empyreumatic odor is not detrimental, it is best to prepare from the crude wood-vinegar distilled wood-vinegar, and work the latter into acetates. Crude lead acetate can, for instance, be prepared in this manner.

If the distilled wood-vinegar should be directly used for the preparation of the various acetates, salts would be obtained which on exposure to the air would turn brown in consequence of the oxidation of the tar-products. However, the behavior of some acetates in the heat is made use of to obtain from them chemically pure acetic acid. While nearly all salts of the organic acids are decomposed at a comparatively low temperature, some salts of acetic acid can without suffering decomposition be heated to almost 750° F. But at this temperature



all the tar products adhering to the salt are completely volatilized or destroyed, so that by recrystallizing the heated mass, salts are obtained which are free from empyreumatic substances, and chemically pure acetic acid can then be made.

*Distilled wood vinegar.*—Since crude wood vinegar always contains tar in solution it is absolutely necessary to get rid of it before neutralizing the vinegar and preparing calcium acetate. High-grade "grey acetate" can only be produced from wood vinegar freed from tar, otherwise "brown acetate" containing at the utmost 67 per cent. calcium acetate is obtained. This separation is effected by distillation, the tar remaining in the still.

For this purpose the crude wood vinegar is subjected to distillation in a simple still heated by steam, whereby about 7 per cent. of tar remains in the still. The distillate, called clear vinegar, still contains small quantities of volatile oils. They are removed mechanically by allowing the clear vinegar to stand, or the latter is separated in vats arranged one after the other in the manner of Florence flasks.

Besides acetic acid the clear vinegar contains wood spirit, *i. e.* a mixture of methyl alcohol, methyl acetate, aldehyde, acetone and allyl-alcohol, and can be separated from it by repeated fractional distillation, whereby the methyl acetate, however, passes over into the woodspirit, thus causing losses of acetic acid. It is, therefore, better to first neutralize the distilled wood vinegar with milk of lime, whereby the greater portion of the methyl acetate is saponified, *i. e.* split into calcium acetate and methyl alcohol, and then distil it. The crude wood spirit is then subjected to rectification and the solution of calcium acetate is evaporated and allowed to crystallize.

This method is obviously inexpedient in so far that distillation takes place twice. The heat used for the first distillation of the crude wood vinegar is therefore lost. Proceeding from this point of view, M. Klar has devised the so-called "three still system". The mixture of vapors appearing in the first distillation of the crude wood vinegar is at once conducted

into milk of lime where calcium acetate which remains in solution is formed from the acetic acid. The vapors again pass into a still filled with milk of lime where any acetic acid which has been carried along is fixed. The vapors pass finally into a cooler where the wood spirit is condensed. Since the boiling point of wood spirit is lower than that of acetic acid, the former does not pass over up to the end of the operation. The cooler may therefore be previously disengaged and the vapors be allowed to escape or be used for other purposes. A special advantage of this method is the complete utilization of the latent heat of the vapors coming from the first still. Moreover the milk of lime in the second and third stills gets to boiling whereby the solution of calcium acetate is at the same time concentrated. The milk of lime in the still has of course to be renewed before it is completely saturated with acetic acid. When working according to this method three advantages are consequently gained in one operation, the crude wood vinegar is freed from tar, the wood spirit is separated and the solution of calcium acetate is concentrated. A solution of calcium acetate of 20 to 35 per cent. is obtained, and a distillate with about 10 per cent. wood spirit.

A great saving in fuel, steam and space is effected by F. H. Meyer's system (German patent, 193,382) of distilling the crude wood vinegar in multiple evaporators in vacuum. It is based upon the fact that all fluids under a decreased air-pressure boil at a lower temperature. Hence if vapors with a lower temperature than the boiling point of the fluid to be evaporated are at disposal for heating purposes, they may be used for distillation by correspondingly decreasing the air-pressure over the fluid to be evaporated.

Freshly-prepared distilled wood vinegar is a colorless, very acid fluid with an empyreumatic odor. On exposure to the air it acquires a brown coloration in consequence of the oxidation of the empyreumatic bodies. Although a number of expedients have been suggested for freeing the distilled wood vinegar from the empyreumatic odor and taste, none of them

have answered the purpose so far as to make it possible to use the vinegar for comestible purposes. The surest proof of the inexpediency of these methods is found in the fact that none of them has been adopted in practice, though by direct conversion of the distilled vinegar, table vinegar could be produced at a very low price.

The distilled wood vinegar may, however, be directly used for technical purposes, for instance, in the preparation of lead acetate, copper acetate, etc. When it is desired to obtain a pure preparation, precaution should be taken to change the receiver of the apparatus in which the crude wood vinegar is distilled when about 80 to 85 per cent. of the total quantity of vinegar which can be obtained has passed over, experience having shown that the last portions of the distilled vinegar are far richer in empyreumatic substances than those passing over first.

Stolze has proposed several methods for the purification of wood vinegar, the most simple and cheapest being to add 5 pounds of finely pulverized pyrolusite to every 100 quarts of vinegar, keeping it at nearly a boiling heat for 6 hours, then digesting it in the same manner with 40 pounds of freshly glowed charcoal pulverized and sifted while hot, and finally distilling off to dryness in a shallow cast-iron still. But on account of its tediousness and the necessarily large consumption of fuel, this process, though frequently modified, has been almost entirely abandoned.

According to Tereil and Château, the wood-vinegar is purified by compounding it, according to its more or less dark color, with 10 or 5 per cent. of concentrated sulphuric acid, whereby the greater portion of the tar separates in 24 hours. By distilling the decanted acid it is obtained almost colorless, but it darkens somewhat on exposure to the air, and by saturation with soda a slightly colored salt is obtained which can, however, be discolored by a small consumption of animal charcoal.

Rothe employs a peculiar method for the purification of



wood-vinegar. The greater portion of tar being separated by standing, the wood-vinegar with an addition of charcoal is rectified from a copper still. The pale yellow watery wood-spirit is caught by itself, and the succeeding clear, but strongly empyreumatic, distillate is pumped into a vat, placed at a considerable height, from which it runs into a purifying apparatus. The latter consists of a cylindrical pipe of stout tinplate. It is about 26 feet high and  $1\frac{1}{4}$  feet in diameter, and is filled with pieces of coke about 0.122 cubic inch in size, which rest upon a heavily tinned iron grate placed about  $1\frac{1}{2}$  feet above the bottom of the pipe. Over this column of coke the wood-vinegar is poured in an uninterrupted fine spray, while in the space between the bottom and the grate a slow current of air heated to  $104^{\circ}$  F. is constantly blown in through a nozzle. The empyreumatic oils mixed with the wood-vinegar are oxidized by the oxygen of the warm air, and, in consequence, the temperature in the interior of the column of coke rises to  $122^{\circ}$  F., and over. The pipe is protected from cooling off by a thick layer of felt. The products of the oxidation of the empyreumatic oils are partially of a resinous nature and adhere to the coke, and partially volatile. The acetic acid running off through an S-shaped pipe on the bottom of the pipe is clear, of a pure acid taste, and suitable for the preparation of all the acetates as well as of acetic acid. The very slight empyreumatic odor disappears by forcing the product through a pipe filled with pieces of animal charcoal free from lime. The vinegar thus obtained is claimed to be suitable for table use. Though a quantity of acetic acid is carried off in the form of vapor by the warm dry current of air, this loss can be prevented by passing the air through another pipe filled with calcined soda or lime.

Experiments have shown that the method above described cannot be recommended for practice, because by the resinous oxidation-products deposits are soon formed upon the pieces of coke which obstruct the free passage of the current of fluid and air, necessitating a frequent renewal of the charge of coke.

Besides a portion of the oxidation-products remains dissolved in the vinegar itself, rendering it for this reason alone unfit for table use.

The only way to prepare perfectly pure acetic acid from the wood vinegar is to fix the acetic acid to strong bases, strongly heat the resulting salts so that all tar substances are volatilized or decomposed, and to separate from the salts thus purified acetic acid by distillation with strong acids. According to this process crystallized acetic acid—the chemically pure preparation—can finally be prepared.

*Production of pure acetic acid from wood vinegar.*—The basic bodies employed in the practice to fix the acids contained in wood vinegar are, according to the object in view, either lime or sodium, the former being used for the preparation of acetic acid suitable for technical purposes, and the latter for that of absolutely pure acetic acid fit for comestible use. In many plants not equipped with the apparatus required for the production of pure acetic acid, the crude wood spirit is distilled off from the wood vinegar and the residue in the still used for the preparation of crude calcium acetate, these two raw products being sold to chemical factories. This course is of special advantage where the charges for transporting the chemicals are very high, the weight of the crude wood spirit and that of the crude calcium acetate being very likely scarcely 10 per cent. of the weight of the wood used. Besides working up the calcium acetate for acetone has also to be taken into consideration.

Since calcium acetate and sodium acetate are the technically most important salts of acetic acid their preparation will be somewhat fully described.

*Preparation of calcium acetate.*—For the neutralization of the crude wood vinegar freed by distillation from wood spirit, burnt and slaked lime is generally used, though as acetic acid is a strong acid and can with ease displace carbonic acid from salts, lime stone *i. e.* carbonate of lime may also be employed for the purpose. The limestone must, however, be quite pure,

especially as free as possible from organic substances, and neutralization has to be effected in large vessels, as the calcium acetate solution foams very much in consequence of the escaping carbonic acid; this drawback is avoided with the use of burnt lime.

The neutralized fluid should be allowed to stand several days so that the tarry substances contained in the wood vinegar can collect on the surface and be removed. It is of importance to only just neutralize the wood vinegar with lime and not use lime in excess, because then a portion of the acid tar products passes already into the layer of tar collecting on the surface and can be separated together with it from the calcium acetate solution. When this has been done the solution is mixed with  $1\frac{1}{4}$  to  $1\frac{1}{2}$  per cent. by volume of crude hydrochloric acid and allowed to rest. The mass which thereby separates on the surface consist chiefly of those substances in which creosote occurs. It is collected and worked by itself for creosote.

The clear calcium acetate solution is evaporated in shallow iron pans, which may be heated by the fire gases escaping from the retort ovens. The tarry substances which separate during evaporation in the form of pitch-like masses are carefully removed. Evaporation is continued till the specific gravity of the hot fluid is  $= 1.116$  or  $15^{\circ}$  Bé. When this point has been reached, the boiling hot, highly concentrated solution of the salt commences, on being further evaporated, to separate crusts of salt. These crusts are removed and completely dried in smaller pans, whilst being constantly stirred. In plants having power at their disposal, evaporation and drying can be effected in one vessel, a circular pan in which a stirrer moves uninterruptedly being used in this case. When, as previously mentioned, the fire gases escaping from the retort ovens are utilized for heating the evaporating pans, there is no danger of the decomposition of the calcium acetate by overheating of the salt mass, since by simply pushing a slide the fire gases can be immediately given another direction. Overheating of the mass in drying is indicated by the



characteristic odor of acetone. While calcium acetate is decomposed at between  $426^{\circ}$  and  $428^{\circ}$  F., acetone being evolved and carbonate of lime remaining behind, the process commences already at about  $302^{\circ}$  F.

It is most expedient to evaporate the calcium acetate solution only to a doughy mass which can be lifted out with a shovel, and to effect the complete drying of this mass upon iron plates forming the bottom of a flat arch and heated by the fire gases escaping from the retort ovens. The temperature in the arches should be so regulated as to never exceed  $302^{\circ}$  F., but the crude salt should be exposed for several hours to this temperature, because by long-continued heating at a lower temperature a great many more tarry substances are destroyed and volatilized than by stronger heating for a shorter time, which besides is accompanied by the danger of decomposing a portion of the calcium acetate.

In heating the evaporating pans by a direct fire there is always danger of overheating. Besides, the pans soon become covered with a crust of calcium acetate, which renders the transmission of the heat very difficult. For this reason round or square pans heated by steam are as a rule used in large modern plants. These pans have a double bottom into which steam is conducted. Copper pans are preferable to iron ones, since the acetate burning to the pans can be more readily removed from copper. The use of such pans is, however, only advisable for the evaporation of solutions already concentrated. For dilute solutions it is better to first concentrate them in multiple evaporators in vacuum, F. H. Meyer's system, German patent 193,382, previously referred to. In this apparatus the solution is brought to 30 to 35 per cent. dry substance and then transferred to the open pans mentioned above.

To avoid the tedious and disagreeable work of completely evaporating and drying the calcium acetate in pans, M. Klar has devised a continuously-working apparatus. It consists of a revolving hollow iron cylinder heated inside by steam or

waste gases. In revolving, the heated cylinder dips into the calcium acetate solution and becomes coated with a thin layer of it, which dries quickly and is removed by scrapers. With this apparatus gray acetate with 80 per cent. can be prepared from acetate solution in one uninterrupted operation. Since, however, the acetate is obtained in the form of a fine, light powder, Klar carries on the drying process only far enough to obtain a product which is no longer sticky. This is dried in a closed band heated by warm air, and at the same time granulated.

The crude gray acetate thus obtained forms a gray odorless mass. It consists of about 80 per cent. calcium acetate and is a commercial article. In addition to calcium acetate it contains calcium butyrate and propionate, as well as certain empyreumatic bodies, and the acetic acid prepared from it also contains butyric acid, propionic acid, etc. Hence this acetic acid cannot be directly used for comestible purposes, but is suitable for most technical uses. Calcium acetate is largely used in print works and in dyeing, and also serves for the preparation of acetone.

*Preparation of Sodium Acetate.*—Sodium acetate in a pure state can be obtained according to several methods which, however, differ from each other only in a certain stage of the operation, the latter beginning always with the neutralization of the wood vinegar freed from wood spirit. For this purpose sodium carbonate is preferably used, as crystallized soda, in consequence of its content of water of crystallization, entails high charges for transport.

Neutralization is effected by adding gradually the sodium carbonate to the wood vinegar, as otherwise the escaping carbonic acid causes strong foaming and the fluid would run over even with the use of a very tall vessel. Enough soda should be added to the wood vinegar for the fluid to contain a very small excess of sodium carbonate, because the sodium acetate crystallizes with greater ease from a slightly alkaline fluid than from a perfectly neutral one.

After adding the soda the fluid is allowed to rest for one day for the separation of the tarry substances, and after removing the latter, the fluid is evaporated in shallow pans, which are heated by the fire gases escaping from the retort ovens or over an open fire. Evaporation is continued until the *hot* fluid shows a specific gravity of  $1.23 = 27^{\circ}$  Bé. The fluid is then emptied into the crystallizing boxes, in which, after the separation of the crystals of crude sodium acetate, remains the mother-lye. The latter is returned to the evaporating pans.

The mother-lye is at the ordinary temperature a saturated solution of sodium acetate, mixed, however, with the bulk of sodium butyrate and sodium propionate contained in the wood vinegar used. When these mother-lyes are continually returned to the evaporating pans, the quantity of sodium butyrate and sodium propionate finally accumulates to such an extent that in cooling the fluid evaporated to specific gravity 1.23, a granular crystal mass is no longer formed, but a soft paste is separated. In this case the fluid in the pans has to be entirely removed and treated as will be described later on.

It is of great importance as regards the purification of the crude crystals to cool the evaporated fluid very rapidly in order to obtain small crystals which retain but little mother-lye. For this purpose oblong sheet-iron crystallizing pans with slightly inclined sides are used. When the contents of the crystallizing vessels have cooled to the ordinary temperature, they form a dark-colored paste of crystals which holds the entire quantity of mother-lye.

To separate the crystals as completely as possible from mother-lye one of two methods may be adopted, namely, draining and washing, or by means of a centrifugal. According to the first method the crystallizing pans are placed in a slanting position, whereby a great portion of the mother-lye runs off and is returned to the evaporating pans. The mass of crystals is brought into a vat with a false bottom, below which is a discharge pipe. When the vat is filled with the mass of crystals, water is poured in. The water dissolves a certain

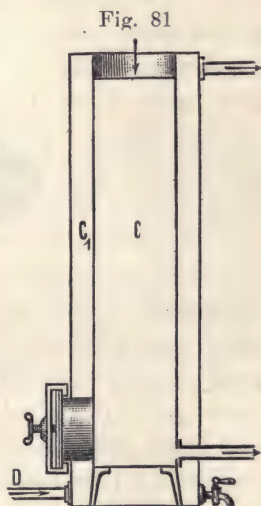


quantity of sodium acetate, and this solution in sinking down displaces the mother-lye, a salt of a quite pale brown color remaining behind.

However, as this method requires considerable time and leaves the salt in a wet state, it is preferable to free the salt from the mother-lye by means of a centrifugal, it being then obtained perfectly dry. In distilling this salt with sulphuric acid, an acid is obtained which to be sure is still empyreumatic, but which can be directly used for many manufacturing purposes.

To obtain pure sodium acetate, the crude salt is dissolved in water by means of steam so that a nearly boiling solution of  $15^{\circ}$  Bé. is obtained, which is then filtered in a hot state through animal charcoal in a filter which can be heated. The filter, Fig. 81, consists of an iron cylinder, *C*, 10 to 13 feet high, enclosed in a somewhat larger iron cylinder *C'*. The inner cylinder is filled with granulated animal charcoal, and steam circulates in the space between the two cylinders. To avoid the necessity of charging a filter in too short a time with fresh animal charcoal, four to six of such filters are arranged in a battery. When the first filter becomes ineffectual, it is emptied, charged with fresh animal charcoal and placed as the last in the battery. Filtration of the hot solution should progress only with such rapidity that a colorless fluid runs off from the last filter. This fluid when rapidly cooled deposits small colorless crystals which after having been freed from mother-lye by means of a centrifugal and dried, pass in commerce as pure sodium acetate.

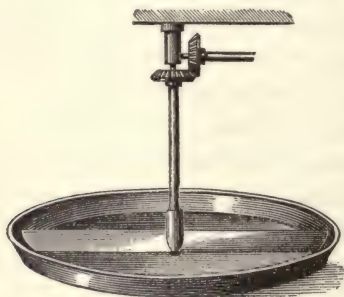
However, even to the salt purified in this manner adhere certain, though only very small quantities, of sodium butyrate



and sodium propionate, and the acetic acid prepared from it contains the corresponding quantities of butyric and propionic acids. The odor of butyric acid is, however, so penetrating that its presence in the acetic acid can be immediately detected by the sense of smell. By rubbing such impure acetic acid upon the palm of the hand, the disagreeable odor of butyric acid becomes conspicuous as soon as the more volatile acetic acid has evaporated.

Hence for the preparation of perfectly pure acetic acid such as is demanded for comestible purposes, a different course has to be adopted which to be sure is somewhat more troublesome than the previously described process but surely accomplishes

Fig. 82.



the object in view. It is based upon the fact that sodium acetate may be heated to nearly  $752^{\circ}$  F. without suffering decomposition, while sodium butyrate and sodium propionate are decomposed and the tarry substances volatilized at a considerably lower temperature.

The salt obtained from the first crystallization purified by washing or by means of a centrifugal is used for this purpose. It is melted in a cast-iron boiler, Fig. 82, about 5 feet in diameter and about 8 inches deep, equipped with a stirrer furnished with two curved blades. The salt at first melts very rapidly in its water of crystallization, yielding the latter with heavy foaming, so that finally a crumbly yellow-brown

mass remains behind which constantly emits tar vapors. The fire under the kettle is kept up uniformly for about one hour and is only sufficiently increased for the mass to melt when no more vapors rise from the latter. The melted mass is lifted from the kettle with shallow shovels and poured upon sheet-iron plates where it congeals to a gray-white cake full of small blisters.

When the operation above described has been correctly carried on, the congealed melt contains sodium acetate, carbonaceous matter and such a small quantity of tarry substances that, when brought into water, it yields a solution of a very pale yellow color. If the heat has been raised too high, a portion of the sodium acetate is also decomposed, acetone being evolved and soda remaining behind. It may sometimes happen that the whole mass takes fire; the latter is extinguished by throwing crude crystals upon it.

The melted mass is dissolved in boiling water. The boiling hot solution, which is colored dark by suspended particles of carbonaceous matter, is filtered through a filter filled with sand and heated by steam, and then quickly cooled in order to obtain small crystals which after treatment in a centrifugal should be perfectly colorless. Since it is next to impossible to heat every portion of the melting mass exactly so long until all the coloring matters have been destroyed, solutions of a yellow color yielding yellow crystals are sometimes obtained. By redissolving these yellow crystals in boiling water and passing the solution through an animal charcoal filter entirely colorless crystals are also obtained.

The sodium acetate thus obtained forms colorless crystals of the composition  $\text{NaC}_2\text{H}_3\text{O}_2 + 3\text{H}_2\text{O}$ , which effloresce on exposure to the air. At the ordinary temperature the salt dissolves in about three times its quantity by weight of water. With an increasing temperature, its solubility becomes much greater and the saturated solution, boiling at  $255.2^\circ \text{F.}$ , contains for 100 parts of water 208 parts of the salt. When heated the salt melts at  $172.4^\circ \text{F.}$ , yields its water of crystallization, and



congeals. It then melts again only at  $606.2^{\circ}$  F., and in a melted state can be heated to between  $716^{\circ}$  and  $752^{\circ}$  F., without suffering decomposition. When heated above this temperature it evolves acetone, becomes readily ignited in the air, and finally leaves a residue consisting of sodium carbonate and coal.

The mother-lyes which in the course of time accumulate in the pan and finally no longer crystallize are evaporated to the consistency of syrup and stored in vats. In a few weeks they are separated from the crude salt and further worked. In most plants this is done by evaporating the lye to dryness and incinerating the residue whereby calcium carbonate mixed with coal remains behind, which is again used for the neutralization of wood vinegar.

When 100 parts of the strongly inspissated lye are mixed with 20 parts by weight of strong alcohol and 70 parts by weight of sulphuric acid are gradually added, a black, oily layer separates on the surface of the fluid. This layer consists of crude acetic, butyric and propionic ethers besides small quantities of formic, valeric and capric ethers and from these raw products all the mentioned acids can be prepared in a pure state.

The purification of the sodium acetate by filtration through animal charcoal is at present only seldom practised, the melting process being more simple to manipulate and yielding better results.

Sodium acetate can also be prepared from calcium acetate by transposition with a soluble sodium salt, the acid of which forms with the calcium an insoluble combination. By mixing, for instance, a solution of calcium acetate with one of sodium sulphate (Glauber's salt), insoluble calcium sulphate is formed and sodium acetate remains in solution. The calcium sulphate (gypsum) is, however, not entirely insoluble and it is therefore far better to effect transposition with the use of sodium carbonate whereby calcium carbonate dissolving with greater difficulty is formed.

*Preparation of acetic acid from the acetates.*—For the preparation of acetic acid in a free state the calcium acetate is decomposed by an acid and the acetic acid separated by distillation. Hydrochloric acid was formerly generally used for the purpose, it having the advantage of forming with the calcium, calcium chloride which is readily soluble in water and in distilling presents fewer obstacles than the calcium sulphate (gypsum) formed by the decomposition of the acetate with sulphuric acid, but the latter is now almost exclusively used, this process being preferable because, on the one hand, the apparatus required for it has been greatly improved and, on the other, the gray acetate with 80 to 82 per cent. acetate furnishes a better and purer raw material. The further manipulation of the acetic acid is then effected in a distilling column which renders it possible to prepare at once from the crude acid, perfectly pure acetic acid and also glacial acetic acid.

*Hydrochloric Acid Process.*—The decomposition of the calcium acetate may be effected by aqueous, as well as by gaseous hydrochloric acid. As previously mentioned, the hydrochloric acid process has been generally abandoned, it being now in use only where brown acetate with about 67 per cent. acetate is to be worked. This product being far more impure is for that reason not suitable for decomposition with sulphuric acid, because the resinous and tarry substances which are present in abundance exert a reducing action upon the sulphuric acid.

The quantity of calcium acetate to be treated is brought into a vat and after pouring the requisite quantity of hydrochloric acid over it, the mass is thoroughly stirred and then allowed to rest for 24 hours. During this time it liquifies and tarry substances separate on the surface. These substances have to be carefully removed before bringing the contents of the vat into a still.

The quantity of hydrochloric acid required for the decomposition of the calcium acetate could very readily be accurately determined if the combinations contained in the salt, which are

decomposed by hydrochloric acid, were exactly known. But this can only be learned from a complete analysis of a sample of the calcium acetate. However, in the practice this troublesome work is generally avoided and the required quantity of hydrochloric acid is determined by pulverizing a portion of the calcium acetate and, after adding to every 100 grammes of salt 90 or 95 grammes of hydrochloric acid, distilling the mass in a small glass still. The distillate is tested for the presence of hydrochloric acid by the addition of solution of nitrate of silver. If after a short time the fluid commences to opalesce or a caseous precipitate is formed in it, hydrochloric acid is present.

A content of hydrochloric acid renders the acetic acid unsuitable for many purposes, and, hence, the use of a small excess of calcium acetate is advisable. When the operation is carefully conducted and especially too rapid distillation connected with squirting of the mass avoided, the acetic acid then obtained contains only traces of hydrochloric acid, and can be freed from them by rectification over some calcium acetate. With the use of crude hydrochloric acid of 1.16 specific gravity, an acid is obtained from the calcium acetate which contains between 47 and 50 per cent of acetic anhydride, and possesses a yellowish color and a slightly empyreumatic odor and taste.

Distillation is effected in a copper still which is protected from the direct action of the fire by an iron shell. The worm may be made of lead and should below be furnished with a U-shaped piece which, when distillation begins, becomes immediately filled with acetic acid and prevents the entrance of air into the worm. By thoroughly washing the worm with water after each distillation it is not attacked by the acetic acid, or only so slightly that the quantity of lead which by these means reaches the acetic acid is insignificant in a product intended for technical purposes.

If rectification of the acetic acid is effected over potassium dichromate instead of over calcium acetate, an acid is to be sure obtained which contains no hydrochloric acid and is



colorless, but has still a very perceptible empyreumatic taste. Since potassium dichromate, 1 to  $1\frac{1}{2}$  lbs. of which has to be used for every 100 lbs. of acid, is quite expensive, its use for the preparation of acetic acid from calcium acetate cannot be recommended, since the resulting acetic acid, on account of its empyreumatic taste, cannot be used for comestible purposes.

The decomposition of the acetate can, as previously mentioned, be also effected with gaseous hydrochloric acid, the advantage of this process being that concentrated acetic acid is at once obtained. The operation is carried on by bringing a sufficient quantity of finely pulverized calcium acetate into vertical retorts which can be heated from the outside. Gaseous hydrochloric acid, previously heated, is conducted through the retorts and the escaping vapors of acetic acid are condensed. However, with the use of this process, the crude acetic acid obtained is very much contaminated with hydrochloric acid, especially towards the end of the operation when the greater portion of the calcium acetate has already been decomposed.

*Sulphuric acid process.*—At present the decomposition of the calcium acetate is, as previously mentioned, almost exclusively effected with sulphuric acid, insoluble calcium sulphate being separated which forms a viscid, pasty mass, and finally becomes solid. In order to attain complete decomposition the mass has to be thoroughly shaken, a work which requires considerable expenditure of power. Gypsum is a bad conductor of heat, and although heat is liberated during the progress of the reaction itself whereby a portion of the acetic acid evaporates, to obtain the last remnants of acetic acid which are tenaciously held by the gypsum, is connected with difficulties. With the use of higher temperatures than attainable with direct firing the mass can to be sure be so far heated that all the acetic acid finally passes over, but a heavy reduction of sulphuric acid then also takes place. For this reason the more modern processes work with the use of a vacuum and steam for heating, far better yields and a purer acid being thereby obtained.

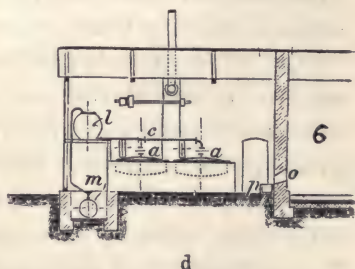
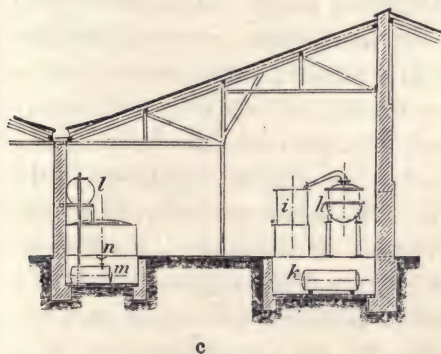
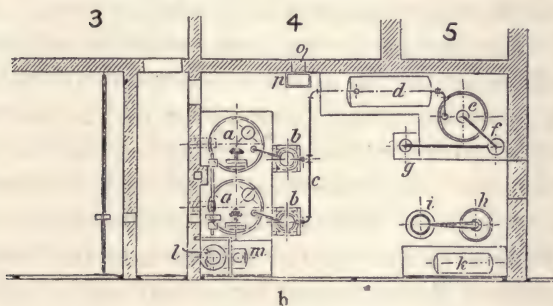
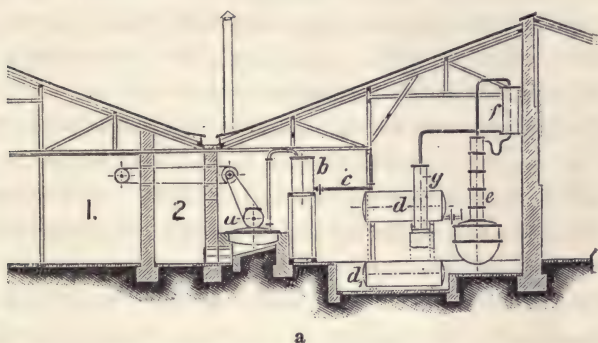
Below a description of an older plant arranged according to Bühler for the sulphuric acid process will first be given, Fig. 83 *a* to *d*.

From the lime kiln the roasted calcium acetate is directly thrown through the funnel *o* into the storage receptacle *p*, which serves also as a measuring vessel for one charge. Decomposition is effected in shallow cast-iron pans *a*, equipped with stirrers, and the covers of which are furnished with man-hole, exhaust pipe, safety valve and inlet for acid. From the reservoir *l*, concentrated sulphuric acid is allowed to run in through a lead pipe conduit. For 100 parts of calcium acetate 60 parts of sulphuric acid are, as a rule, allowed. Decomposition at first progresses by itself and about  $\frac{1}{6}$  of the acetic acid present distils over. Slight heating then becomes necessary. The stirrers must be kept constantly in operation. The acetic acid vapors pass from *a* into a clay condenser *d*, and run through *c* into a storage-reservoir *d* of clay; all other materials would in a short time be destroyed.

The crude acid still contains impurities, such as sulphurous acid, traces of sulphuretted hydrogen, etc., which by its partial decomposition the sulphuric acid has yielded together with the tar of the crude acid; besides it contains resinous and tarry substances and coloring matter which are removed by rectification over potassium chromate.

For this purpose acid is allowed to run from the reservoir *d* into the still, and after the addition of water is rectified. The apparatuses used at the present time at once furnish a product of 99 per cent. and more. In the illustration, *e* is the column, *f* a pipe condenser with return pipe, and *g* a condenser for the acid. For the preparation of vinegar for comestible purposes the acid is again rectified in the still *h*, with the addition of potassium chromate, a perfectly clear product without detrimental odor being obtained from the clay condenser *i*. The still *h* may be of enameled cast iron and is provided with a heating jacket. The movement of the fluid is effected by means of compressed air and the monte-jus *m*, *d*, and *k*.

FIG. 83.



1. Engine-house ; 2. Fire passage ; 3. Boiler-house ; 4. Lime Kiln ; 5. Storage for calcium acetate ; 6. Lime kiln.

The preparation of glacial acetic acid is effected by decomposing the sodium salt by means of sulphuric acid. By the introduction of sodium sulphate (Glauber's salt) into the cal-



cium acetate solution, the latter is converted into sodium acetate solution, saturation being attained when a clear filtered sample no longer yields a precipitate of calcium sulphate on the further addition of sodium sulphate.

The solution is drawn off from the sediment and the latter lixiviated until exhausted. Concentration to a specific gravity of 1.3 is effected in directly heated boilers. The excess of sodium sulphate crystallizing out is brought into perforated baskets from which the mother-lye again runs into the boilers. It is then allowed to settle and clarify for 8 to 10 hours when it is drawn off. The sediment consists of admixtures of the raw materials which have become insoluble, tar and other constituents. In coolers or crystallizing vessels the greater portion of the sodium acetate is deposited in three to five days, and the crude salt is frequently directly sold. The mother-lye is drawn off, is again concentrated, crystallized, and so on until exhausted. The residue is then evaporated and heated to a red heat in order to obtain sodium carbonate, or heated to melting to remove the tar, the sodium acetate thus obtained being separated from the coal by dissolving in water. The tarry admixtures, tar oils of various kinds, adhere with great tenacity to all the products of distillation, even the crystals first obtained being not perfectly pure. They are purified by again dissolving them, concentrating the solution and crystallizing. The crystals are then melted in an iron boiler in the water of crystallization and, after evaporating the latter, heated until melted the second time. The salt is now anhydrous and great care is required to prevent it from burning. It is then decomposed by concentrated sulphuric acid in glass retorts in a sand bath. For 92 parts salt 98 parts sulphuric acid are used. From the distillate the glacial acetic acid separates, on cooling, in the form of crystals.

At the present time the *vacuum process* is generally employed, it yielding at once a highly concentrated acetic acid. The apparatus used for this purpose as constructed by the firm of J. H. Meyer, at Hanover-Hainholz, Germany, consists of cast

iron boilers which according to the size of the plant, have a capacity of from 240 to 3300 lbs. of calcium acetate. Each boiler is equipped with a stirrer. The charge is introduced through a manhole in the cover and the boiler is emptied, as a rule, through an aperture in the bottom through which the gypsum can be pushed out by the stirrer.

When the gray acetate is uniformly distributed in the boiler, the calculated quantity of sulphuric acid is allowed to run in. Decomposition then commences, so much heat being thereby liberated that a large portion of the acetic acid distils over without the use of a vacuum. When distillation slackens, vacuum is applied and distillation carried on to the end, the bottom of the boiler being at the same time heated by steam. From 220 lbs. of acetate and 132 lbs. of sulphuric acid, 165 lbs. of crude acetic acid with 80 per cent. acid are under normal conditions obtained. The crude acid contains small quantities—0.005 to 0.05 per cent.—of sulphurous acid.

For the preparation of acid intended for technical purposes only, the crude acetic acid is further purified by subjecting it once more to distillation in more simple copper stills generally provided with a worm silvered inside. For the preparation of high-graded, entirely pure acid and of vinegar essence suitable for comestible purposes, the crude acetic acid is decomposed in a column apparatus.

In order to finally prepare chemically pure vinegar (99 to 100 per cent.) from that fraction which contains 96 to 98.5 per cent. of acetic acid, the distillate is further treated with potassium permanganate for the oxidation of contaminating admixtures still present, and then distilled from the "fine acid apparatus". This apparatus has a still of copper, but a head and worm of silver to prevent contamination by copper acetate. Generally the first and last runnings only are removed, the middle running being perfectly pure acetic acid.

Glacial acetic acid, of the highest concentration, *acidum aceticum glaciale*, can also be prepared as follows: Freshly dehydrated sodium acetate is distilled with concentrated sulphuric

acid, or water is withdrawn from high-graded acetic acid by rectifying it over fused calcium chloride.

In the first case  $9\frac{1}{2}$  parts by weight of sulphuric acid are slowly poured upon 100 parts by weight of sodium acetate free from water to prevent the escape, without being condensed, of a portion of the acetic acid vapors evolving with great vigor. Heat is applied only after the introduction of the total quantity of sulphuric acid, and the first four-fifths of the distillate are caught by themselves, because the last portion of it has an empyreumatic odor, while the first portions contain only sulphurous acid which is removed by rectifying the acid over potassium dichromate.

According to the second process glacial acetic acid may even be prepared from 50 per cent. acetic acid by distilling the latter with fused (anhydrous) calcium chloride. The glacial acetic acid thus obtained contains considerable quantities of hydrochloric acid. It is freed from it by rectification over anhydrous sodium acetate, a still with a silver head and worm being used, and the precaution taken not to cool the worm too much as otherwise the acetic acid might crystallize in it.

The calcium chloride containing water which remains behind is dehydrated by heating in shallow vessels and then heated to red hot fusion, this being necessary for the destruction of all organic substances present. The preparation of glacial acetic acid should, if feasible, be undertaken in the cool season of the year. The distillate running from the condenser is collected in stone-ware pots which are covered and exposed to a low temperature, the greater portion of the fluid congealing thereby to a crystalline mass. The position of the pots is then changed so that the portion which has remained fluid can run off; this is added to the next rectification over calcium chloride. By placing the pots in a heated room the anhydrous acetic acid is melted and then filled in bottles. Glacial acetic acid thus prepared will stand the test usually applied in commerce. It consists in bringing the acetic acid together with lemon oil. Anhydrous acetic anhydride dissolves lemon oil



in every proportion, but in the presence of even a very small quantity of water solubility decreases in a high degree. Another commercial test consists in compounding the acetic acid as well as the glacial acetic acid and the diluted acid, with solution of potassium permanganate till it appears raspberry red; pure acid remains permanently red, while if empyreumatic substances are present, the red color disappears rapidly.

However, an acid free from empyreumatic substances, but containing sulphurous acid, may also exert a discoloring effect upon the potassium permanganate solution. It is, therefore, advisable before making the test for empyreumatic substances to test the acid for sulphurous acid. This is done by compounding the acid with potassium permanganate, allowing it to stand until discolored and then adding barium chloride solution; sulphurous acid, if present, has now been changed to sulphuric acid, the fluid is rendered turbid by barium chloride, and after standing for some time a white precipitate is separated. Such acid should again be rectified.

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## CHAPTER XXV.

### ACETATES AND THEIR PREPARATION.

The acetates, particularly those of calcium, potassium, sodium, barium, lead, copper, aluminium and iron are extensively used in the industries and are produced in large quantities. Calcium acetate and sodium acetate are as previously explained initial products for the preparation of acetic acid and acetone, for which barium acetate may also be used.

All the acetates are more or less readily soluble in water. By the addition of sulphuric acid the acetic acid is liberated without perceptible evolution of gas. The solutions of the acetates are precipitated by nitrate of silver; the precipitate

of acetate of silver is crystalline and soluble in 100 parts of water.

When mixed with equal parts of alcohol and double the weight of sulphuric acid, acetic ether is evolved, which is recognized by its characteristic fruit-like odor.

Ferric chloride imparts to the very dilute aqueous solution an intense bright-red color.

From other similar combinations the acetates are distinguished by yielding acetone when subjected to destructive distillation, and marsh-gas when distilled with caustic potash. When heated with arsenic the very peculiar and disagreeable odor of cacodyl is evolved.

*Potassium acetate*,  $\text{KC}_2\text{H}_3\text{O}_2$ . Dry potassium acetate forms a snow-white, somewhat lustrous, not very heavy, laminate or foliated crystalline, pulverulent mass. It has a warming, slightly pungent salty taste, and its odor is not acid. It turns red litmus paper slightly blue, but does not redden phenolphthalein. It rapidly absorbs moisture from the air and deliquesces. At the ordinary temperature it is soluble in about  $\frac{1}{2}$  part of water or  $1\frac{1}{2}$  parts of alcohol. Boiling water dissolves eight times the quantity of its weight of potassium acetate, such a solution boiling at  $329^\circ \text{F}$ . The dry salt when triturated with iodine yields a blue mixture, the latter giving with water a brown solution.

When heated, potassium acetate melts, without suffering decomposition, at  $557.3^\circ \text{F}$ ., and on cooling congeals to a radiated crystalline mass. On heating to  $780^\circ \text{F}$ . acetic acid escapes and, after incineration, potassium carbonate colored gray by coal remains behind.

By adding ferric chloride solution to potassium acetate solution, a liquid of a blood-red color is obtained which besides potassium chloride contains ferric acetate. By heating the solution to boiling a red precipitate of basic ferric acetate is separated, the supernatant liquid becoming colorless, provided sufficient potassium acetate was present and the ferric chloride solution did not contain too much hydrochloric acid.

Potassium acetate is prepared by bringing into a vat or boiler purified wood vinegar and adding potash in small quantities. The liquid foams, and the tarry substances that separate on the surface are removed by means of a perforated ladle. The addition of potash is continued till the solution is neutralized when it is allowed to settle. The clarified solution is then evaporated to dryness in an iron pan, the tarry substances appearing during this operation being removed. When the product is dry, the fire is increased and the salt melted in the water of crystallization. When the mass has acquired a butyaceous appearance, the fire is withdrawn and the salt allowed to cool, otherwise it would change to potassium carbonate. When cold the melt is again dissolved in water, filtered and further worked.

Another method of producing potassium acetate is by decomposing normal acetate of lead (sugar of lead) with pure carbonate or sulphate of potassium. To detect the presence of lead it should be tested with sulphuretted hydrogen, which in the presence of this metal produces a slightly brown precipitate. To obtain a pure product the decanted liquid is treated with sulphuretted hydrogen, and, after separating from the precipitate and adding a small quantity of acetic acid, is evaporated in a stone-ware vessel.

Chemically pure potassium acetate is prepared by bringing 400 parts of 30 per cent. acetic acid into an acid-proof enameled boiler and gradually introducing 138 parts of pure potassium carbonate or 200 parts of potassium bicarbonate until the solution is finally neutral or shows but a slight alkaline reaction. Solution has finally to be assisted by heating.

The solution is then acetified with acetic acid and evaporated to a small volume on a steam-bath or better over an open fire. Some acetic acid is next added and the mass is then evaporated till it is dust-dry, being constantly stirred with a porcelain spatula during this operation. The resulting dust-dry, crumbly powder is brought into dry hot vessels, which are immediately closed with corks and the latter are coated with paraffine.



Too strong heating should be avoided, otherwise decomposition takes place, acetone being formed.

Pure potassium acetate should dissolve in two parts of water and the solution must not redden phenolphthalein, otherwise it contains more than traces of potassium carbonate. A 5 per cent. aqueous solution should not be rendered turbid or colored (presence of metals) by hydrogen sulphide, and not altered by barium nitrate after the addition of dilute nitric acid, otherwise it is contaminated with sulphate.

*Potassium acid acetate or potassium diacetate*,  $\text{KC}_2\text{H}_3\text{O}_2\text{C}_2\text{H}_4\text{O}_2$ , is formed by evaporating a solution of the neutral salt in excess of acetic acid; it crystallizes by slow evaporation in long, flattened prisms. It is very deliquescent and decomposes at  $392^\circ\text{F}$ ., giving off crystallizable acetic acid.

*Sodium acetate*,  $\text{NaC}_2\text{H}_3\text{O}_2$ .—The manner of preparing this salt in the manufacture of wood vinegar has already been described. It can be obtained in a manner similar to that of the potassium salt by dissolving carbonate of soda in acetic acid, evaporating the solution, and setting the liquid aside to crystallize. The crystals form large, colorless, oblique rhombic prisms. Their composition is  $\text{NaC}_2\text{H}_3\text{O}_2 + 3\text{H}_2\text{O}$ ; they are soluble in 3 parts of cold, in a less quantity of boiling, water, and in 5 of alcohol.

The taste of sodium acetate is cooling and saline. When exposed to dry air it loses its three equivalents of water, but regains them in a moist atmosphere. After being melted it is deliquescent and takes up 7 equivalents of water. It becomes a liquid, supersaturated solution which crystallizes, with evolution of heat, immediately after a fragment of dry or crystallized sodium acetate is thrown into it.

Sodium acetate is used for the preparation of acetic acid, acetic ether, and in medicine. It has also been recommended for the preservation of animal and vegetable tissues, it being used in the form of a powder in place of common salt.

*Ammonium acetate, neutral acetate of ammonia*,  $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ .—This substance is obtained by neutralizing acetic acid with

carbonate of ammonia, or better, by saturating glacial acetic acid with dry ammonia gas. It is very difficult to obtain in the crystalline form on account of its aqueous solution giving off ammonia when evaporated, thus becoming converted into the acid salt. When subjected to dry distillation ammonia gas escapes first; above  $330^{\circ}$  F. there is formed, besides water, chiefly acetamide ( $C_2H_5NO$ ), a white crystalline body which is also formed, besides alcohol, on heating acetic ether with liquid ammonia in a closed vessel to about  $266^{\circ}$  F.

In medicine ammonium acetate has long been used as a diaphoretic.

*Calcium acetate*,  $Ca (C_2H_3O_2)_2$ . The preparation of calcium acetate has already been described under wood vinegar.

The crystals of the pure salt form white acicular prisms which effloresce in the air and are soluble in water and in alcohol; they have a bitter, salty taste. They are decomposed by heat into acetone and calcium carbonate. A mixture of this salt and of potassium oxalate gives, on heating, propylene ( $C_3H_6$ ), while a mixture of carbonates remains behind. By destructive distillation of equal equivalents of acetate and benzoate of calcium, acetophenone ( $C_8H_8O$ ) is obtained, which by treatment with nitric acid is converted into nitro-acetophenone ( $C_8H_7NO_3$ ). By heating the latter with zinc-dust and soda-lime, Emmerling and Engler claim to have obtained artificial indigo-blue. But the quantity of the latter thus obtained is always very small, and it appears to be very difficult to ascertain the precise condition under which the transformation takes place.

*Barium acetate*,  $(C_2H_3O_2)_2Ba + 1\frac{1}{2} H_2O$ , finds at present extensive application in the industries, especially in the preparation of acetone, as it is decomposed at a lower temperature than calcium acetate, and leaves a residue—barium carbonate—which can again be used for the preparation of the acetate, while the residue from calcium carbonate is of no value.

Barium acetate is prepared from the mineral witherite in a manner similar to calcium acetate, the only difference being as

to whether the witherite is used in lumps or in the form of powder. In the first case the wood vinegar is allowed to act for some time upon the witherite by allowing it to run over the mineral, or after filling several vessels (a battery) with the witherite, dissolving it by conducting gaseous acetic acid through the vessels. Provisions must of course be made for carrying off the carbonic acid that is evolved.

The use of witherite in the form of powder instead of in lumps, is more advantageous; but as the powder, in consequence of its own specific gravity has a tendency to settle on the bottom, a powerful mixing and stirring apparatus has to be provided. When used in the form of a fine powder the witherite must be sifted upon the surface of the fluid containing the acetic acid, as it readily forms lumps, which drop to the bottom and are dissolved only with difficulty. As the evolution of carbonic acid is by no means tumultuous the vessels used can be kept quite full.

The barium acetate solution obtained by either method may be neutral but should never show an acid reaction. It is passed through a filter press and then evaporated in the usual manner, care being taken not to let it boil up; for this reason it is advisable to effect evaporation in a vacuum.

Crystallization is effected in the customary manner. The resulting crystals are freed from the mother-lye and, if not colored too dark (by empyreumatic substances), can be immediately used. Purification is effected by recrystallization, dissolving the crystals in water, by treatment with animal charcoal, china clay or bole, but best by a small addition of sulphuric acid, whereby barium sulphate is formed which falls to the bottom, carrying with it the empyreumatic substances. After settling the clear liquor is drawn off or the mixture is passed through a filter-press. The clear solution is evaporated in the customary manner and, after crystallization, the mother-lye is separated and the crystals, if necessary, are once more purified. The mother-lyes are utilized by adding them to the solutions first obtained. If they contain too many foreign



substances, they are evaporated to dryness, dried, ignited and used for solution in acetic acid.

According to another method barium acetate is prepared by allowing equivalent quantities of barium chloride and sodium acetate to act upon each other. For this purpose the adequate quantity of barium chloride is dissolved in water so that, if feasible, an oversaturated solution is obtained, and to this solution, whilst boiling vigorously, sodium acetate in fine crystals is gradually added. During this reaction the contents of the boiler must constantly be kept boiling vigorously. Decomposition progresses quite rapidly. The sodium chloride which is separated is removed and the progress of reaction watched by taking samples. When decomposition is complete, the contents of the boiler are allowed to settle, and the solution drawn off from the sediment is crystallized. The crystals are then separated from the mother-lye and the last remnants of the latter removed either by suction or by means of a centrifugal, the crystals being at the same time washed with water and, if required, recrystallized from pure water. The mother-lye always contains common salt and is again utilized for dissolving the barium chloride.

The crystallized barium acetate obtained at the ordinary temperature contains 1 molecule of water, while that crystallized at 32° F. possesses 3 molecules of water and is amorphous with lead acetate. On exposure to the air the crystals effloresce and the solutions show an alkaline reaction. The salt is soluble in 1.5 parts of cold, and 1.1 parts of boiling water, and in 67 parts of boiling, and in 100 parts of cold, alcohol.

*Strontium acetate.*—This salt is prepared in a manner similar to that of the preceding. The crystals obtained at 32° F. contain 5 equivalents of water and those at 59° F. 1 equivalent.

With strontium nitrate it gives a double salt forming beautiful crystals which contain 3 equivalents of water. On heating they first yield their water of crystallization and then detonate, a beautiful purple flame being formed.

*Magnesium acetate* is prepared by dissolving *magnesia alba*

or *usta* in acetic acid. It crystallizes with difficulty and is readily soluble in water and spirits of wine. Only a very small portion of the solution is decomposed by ammonia. By destructive distillation it yields acetic acid, while magnesia remains behind.

*Aluminium acetate*.—Large quantities of aluminium acetate are used under the name of *red mordant* in dyeing, as well as for water-proofing tissues.

Three combinations of aluminium with acetic acid are known, namely, normal  $\frac{2}{3}$  aluminium acetate, and two basic acetates which according to their content of acetic acid it is customary to distinguish as  $\frac{2}{3}$  and  $\frac{1}{3}$  aluminium acetates.

The *normal* or  $\frac{2}{3}$  *aluminium acetate*,  $\text{Al}_2(\text{CH}_3\text{CO}_2)_6$  is obtained by decomposing aluminium sulphate with barium acetate, or by dissolving aluminium hydrate in the calculated quantity of acetic acid. It is only known in a liquid form. When evaporated even at below  $100^\circ \text{F}$ . it is decomposed to basic acetates of different compositions. In boiling the solution insoluble basic aluminium acetate is separated, the composition of which is not yet accurately known.

The *basic aluminium*  $\frac{2}{3}$  *acetate*,  $\text{Al}_2(\text{OH}_2)(\text{CH}_3\text{CO}_2)_4$  is obtained by dissolving aluminium hydrate in the calculated quantity of acetic acid. By evaporating the solution at below  $100^\circ \text{F}$ ., it is reduced to a hornlike mass which gives a clear solution with water, especially if the latter has been acidulated\* with acetic acid. By heating above  $100^\circ \text{F}$ . insoluble basic aluminium acetate is separated.

The *basic aluminium*  $\frac{1}{3}$  *acetate*,  $\text{Al}_2(\text{OH}_4)(\text{CH}_3\text{CO}_2)_2$  separates on heating or evaporating the solutions of the abovementioned combinations.

For the preparation of aluminium acetate in larger quantities, aluminium hydrate is seldom used as the initial material, aluminium sulphate or ordinary alum being generally decomposed by means of a carbonate, lead acetate or calcium acetate. The aluminium hydrate thus obtained is then dissolved in acetic acid.

Aluminium sulphate is as a rule preferred to alum, it being cheaper and can also be obtained free from iron, which is of great importance as regards the use of aluminium acetate in Turkey red dyeing (for alazarine colors) as iron imparts a brown tinge to the red.

Aluminium acetate is generally prepared by dissolving 30 parts by weight of aluminium sulphate in 80 parts of cold water, then adding 36 parts by weight of 30 per cent. acetic acid (of 1.041 specific gravity), and introducing into this mixture, whilst stirring constantly, 13 parts by weight of whiting triturated with 20 parts by weight of water.

Decomposition does not proceed as smoothly as would appear from the chemical equation; it must be effected so that heating of any kind is excluded.

By reason of the evolution of carbonic acid the decomposition-vessel must be of such a size that it is filled only two-thirds full. It should be furnished with a stirrer to keep the heavy whiting suspended, and thus cause uniformity of decomposition.

The whiting should be intimately stirred together with the water and in order to retain admixed impurities such as sand, straw, etc., poured in small quantities through a fine-mesh sieve. When all the whiting has been introduced the stirrer is kept running for 5 or 6 hours longer, and the mass is then allowed to stand quietly. Since decomposition is not completely finished in 24 hours, it is accelerated after that time by again running the stirrer for six hours, when the mixture is brought into a filter-press and the heavy solution collected by itself. The press-cakes are thoroughly washed and the wash-water is used for dissolving fresh portions of aluminium sulphate. The cakes remaining in the frame of the filter-press may, if pure white, be dried and sold as gypsum; otherwise they can be utilized as fertilizer.

In place of whiting some manufacturers use sodium bicarbonate; 667 parts by weight of aluminium sulphate are dissolved in 15000 parts by weight of warm water. Into this



solution are introduced very slowly and whilst constantly stirring, 504 parts by weight of sodium bicarbonate, the bicarbonate product of the manufacture of ammonia-soda being suitable for the purpose. Alumina in a gelatinous form is precipitated. It is allowed to settle, washed free from salt and pressed to a weight of 1750 parts. 200 parts by weight of the gelatinous aluminium hydrate are dissolved in 150 parts by weight of 50 per cent. acetic acid. One liter of the mordant thus obtained contains 35.3 grammes of alumina. In place of sodium bicarbonate, ammonia-soda may be used in the proportion of 1 part by weight of aluminium sulphate to 3.6 parts by weight of soda.

Many manufacturers prefer to effect the decomposition of the aluminium sulphate with lead acetate instead of with whiting or sodium carbonate. Since lead sulphate is insoluble the decomposition of the aluminium sulphate solution is more perfect. In practice it is found advantageous to employ equal parts of aluminium sulphate and acetate of lead, or even a rather less quantity of the latter. The aluminium sulphate is dissolved in boiling water, and the powdered lead acetate added to the solution. About one-tenth of crystallized carbonate of soda, or a little carbonate of lime, is added to the alum to combine with the free acid. The three following receipts serve to indicate the proportions employed :—

I. Dissolve 100 pounds of aluminium sulphate in 50 gallons of boiling water, and add 10 pounds of acetate of lead in fine powder, stirring the mixture well at first, and likewise several times during cooling.

II. Dissolve 100 pounds of aluminium sulphate in 50 gallons of boiling water, add slowly 10 pounds of crystallized carbonate of soda, and then stir in 50 pounds of acetate of lead in powder.

III. Dissolve 100 pounds of aluminium sulphate in 50 gallons of boiling water, and add in small portions 6 pounds of crystallized carbonate of soda, and then stir in 50 pounds of acetate of lead, in powder, as before.

The cheapest method of preparing aluminium acetate is from calcium acetate and aluminium sulphate, the principal condition being not to use a too tarry gray acetate as otherwise the final product turns out too dark and can be decolorized only with difficulty. Calcium acetate contains, as a rule, a larger quantity of calcium carbonate, and this quantity has previously to be accurately determined in order to calculate the quantity of aluminium sulphate required. In practice this calculation is avoided by having always on hand clear solutions of both salts and, when an excess of one or the other salt is found in the aluminium acetate solution, correcting it by the addition of one or the other solution, so that a pure aluminium acetate solution results.

As a rule 100 parts by weight of calcium acetate and 70 parts by weight of aluminium sulphate are dissolved, each by itself, in sufficient water to obtain solutions of 5 to 6° Bé. The solutions are filtered and after bringing the separate solutions into the vessels located over the precipitation vat, precipitation is effected. On account of the evolution of carbonic acid, the vat should not be kept too full.

When a determined quantity of fluid has been consumed and evolution of gas has ceased, a small quantity of the fluid in the vat is filtered in a small glass cylinder, the filtrate divided into two halves, one of which is tested with barium chloride solution for sulphuric acid, and the other with ammonium oxalate or sulphuric acid for lime. If in one or the other case a white precipitate is formed enough of one or the other clear solution previously mentioned is added until after taking other samples no precipitate or turbidity is formed in them; the aluminium acetate solution then contains only slight traces of calcium acetate or aluminium sulphate.

The contents of the precipitation-vat are passed through a filter-press and the residue is washed. The cakes of gypsum are not suitable for fertilizing purposes; by adding air-slaked lime and ashes they may be utilized for repairing roads, etc.

*Manganese acetate*,  $\text{Mn}(\text{C}_2\text{H}_3\text{O}_2)_2$ . This substance is pre-

pared by dissolving freshly precipitated manganous carbonate ( $\text{MnCO}_3$ ) in heated acetic acid, evaporating the solution and crystallizing. The crystals are rhombic prisms, and occasionally in plates of an amethystine color; they are permanent in the air, soluble in alcohol, and in about three times their weight of water.

On a large scale this salt is manufactured by precipitating a solution of manganous sulphate \* by one of lime acetate and agitating the liquor to decompose the whole of the manganese salt.

It sometimes happens that a portion of the manganese salt is not acted upon by the acetate of lime, and in this case a concentrated solution of acetate of lead is employed towards the end of the process to effect complete decomposition. The mixed precipitate of sulphate of lime and lead is filtered off, and the filtrate evaporated and crystallized. The best acetate of manganese is made by adding to 4 parts of manganous sulphate dissolved in 3 parts of water, 7 parts of crystallized acetate of lead dissolved in 3 parts of water, agitating the solution, and drawing off the clear liquor for use.

Acetate of manganese is used in dyeing and calico-printing to give a brown color to fabrics. Its principle of action depends upon the further oxidation of the manganese.

*Iron acetate.*—Acetic acid combines with ferrous oxide ( $\text{FeO}$ ) as well as with ferric oxide ( $\text{Fe}_2\text{O}_3$ ), but only the ferrous acetate crystallizes in small greenish-white needles, very prone to oxidation, while ferric acetate is a dark, brownish red, uncrystallizable liquid, of powerful and astringent taste. Both salts dissolve freely in water, and are of importance for dyeing and calico-printing.

*Ferrous acetate,  $\text{Fe}(\text{C}_2\text{H}_3\text{O}_2)_2$ . Black mordant. For dyeing*

\* Manganous sulphate is prepared by mixing the dioxide (pyrolusite) with half its weight of concentrated sulphuric acid and heating in a Hessian crucible until no more vapors escape. The residue is dissolved in water, filtered, and allowed to crystallize at an ordinary temperature. The solution of the salt when decomposed with crystallized soda gives a precipitate of manganous carbonate.



purposes this salt is prepared by dissolving wrought-iron turnings in wood-vinegar, care being had that some iron remains undissolved, as otherwise the salt, on exposure to the air, is gradually partly converted into the ferric salt. This oxidation proceeds, however, but slowly, the empyreumatic substances contained in the wood vinegar rendering the conversion rather difficult. The pure salt oxidizes with great rapidity. For commercial purposes this compound is manufactured as follows: Into a large wooden vat or into barrels a quantity of iron turnings, hoops, or nails are introduced, and hot crude wood-vinegar, freed by distillation from wood-spirit, is poured upon them. During the solution of the iron much tarry matter separates, which is skimmed off, and the solution is frequently agitated to free it as much as possible from the tar. After 24 hours the solution is drawn off. The iron being entirely coated with tar so that it can not be again attacked by the wood-vinegar, it is taken from the vat and the tar ignited. The iron is freed from the oxide formed by sifting and can be again used. The solution thus obtained shows  $13^{\circ}$  or  $14^{\circ}$  Bé.

The pure salt is obtained by dissolving iron in acetic acid or by double decomposition from ferrous sulphate (14 parts) and lead acetate (19 parts); and cheaper, but less pure, from ferrous sulphate and calcium acetate.

If crude calcium acetate instead of wood-vinegar is to be used in the preparation of this salt, a solution of the calcium acetate of specific gravity 1.08 is mixed with half its weight of ferrous sulphate dissolved in  $2\frac{1}{2}$  times its weight of water. On agitating the mixture, the decomposition is rendered complete, the clear liquor which is siphoned off after the subsidence of the sulphate of lime showing  $13^{\circ}$  Bé. It is kept in a closed barrel in which is hung a bag containing a quantity of iron turnings.

In some factories the ferrous acetate is manufactured by decomposing the carbonate of iron ( $\text{FeCO}_3$ ) with lead acetate; lead carbonate precipitates, and the blackish supernatant liquor is the acetate of iron in a very pure state. It is kept from ox-

idizing by immersing in it some bright iron filings. The lead salt formed repays the cost of the manufacture of the acetate.

Solution of ferrous acetate is used as a mordant by dyers, for staining wood and leather and in the manufacture of ink. The commercial article generally shows a specific gravity of 1.10 (12° B.).

On account of the avidity with which ferrous acetate absorbs oxygen, it is of great value as a reducing agent. It is, for instance, used in the preparation of aniline from nitrobenzole and for similar reducing processes.

*Neutral ferric acetate, sesquiacetate of iron,  $\text{Fe}(\text{C}_2\text{H}_3\text{O}_2)_3$ .*—For technical use this combination is obtained by dissolving wrought-iron in wood-vinegar so that it has a chance to oxidize in the air. For this purpose wood-vinegar is poured over iron turnings in a vat, and after drawing off the solution in a few days, the iron is for some time left to the action of the oxygen of the air. It quickly oxidizes, and by pouring back the solution and several times repeating the drawing off and pouring back, a quite concentrated solution of dark red brown, nearly black color is in a short time obtained. Heat must not be employed in the preparation of this salt, as in such case it readily decomposes.

Neutral ferric acetate may be obtained in the pure state by decomposing a solution of lead acetate by adding ferric sulphate in slight excess. In the course of 24 hours the excess of ferric sulphate precipitates as a basic salt. It is also produced, though more slowly, by dissolving ferric hydrate or ferric carbonate obtained by precipitation, in strong acetic acid. This method occupies more time, but affords better guarantees for the purity of the compound.

By dissolving one part of nitric acid or aqua regia, precipitating the solution with ammonia and dissolving the washed ferric hydrate in 10 parts of acetic acid of 1.042 specific gravity, and evaporating the solution at between 140° and 176° F. an amorphous salt soluble in water and alcohol remains, which is, however, not neutral, as it contains only two instead of 3

equivalents of acetic acid for 1 equivalent of ferric oxide. By dissolving this amorphous salt in acetic acid and exposing the dark red solution to a low temperature, the neutral salt crystallizes out in hydrated, lustrous, dark red laminæ.

On heating the strongly diluted solution of this salt to nearly the boiling point its color becomes more intense and it evolves a distinct odor of acetic acid without, however, producing a precipitate. The salt has nevertheless become more basic, and an addition of any soluble sulphate or even of free sulphuric acid immediately precipitates the whole of the iron as insoluble basic ferrous sulphate. By heating, however, the dilute solution of the pure acetate to boiling, it disengages acetic acid and separates a basic salt, which, if boiling be continued, also loses its acid so that ferric hydrate remains behind. The properties of this hydrate differ, however, from those of ordinary ferric hydrate, it dissolving in concentrated hydrochloric acid only by long-continued digestion or boiling, and is scarcely attacked by boiling concentrated sulphuric acid. In acetic acid or dilute nitric acid it dissolves, however, to a red fluid, transparent to transmitted, but opaque to reflected, light. By adding the slightest quantity of a sulphate or of concentrated nitric or hydrochloric acid, a granular precipitate is formed, which, however, redissolves on diluting the fluid with water. If a solution of ferric acetate is heated in a closed vessel to 212° F. for a few hours, the fluid seen by reflected light appears opaque and opalescent. It has also lost its metallic taste and no longer shows the other reactions of ferric salts, *i.e.*, addition of ferrocyanide produces no precipitate nor does the sulphocyanide augment its red color. A trace of sulphuric acid or any alkaline salt suffices to precipitate the whole of the iron in solution as ferric hydrate, of red color, which is totally insoluble in all acids at an ordinary temperature; dilute mineral acids do not, however, produce a similar precipitate. It is remarkable that this ferric hydrate dissolves in water to a dark red fluid which can be again precipitated by concentrated acids or alkaline salts (Péan de St. Giles).



From the iron acetates the iron is precipitated as black ferrous sulphide by sulphuretted hydrogen.

With ferric nitrate, ferric acetate yields a crystallizable double salt,  $\text{Fe}(\text{C}_2\text{H}_3\text{O}_2)_2\text{NO}_3 + 3\text{H}_2\text{O}$ , the solution of which decomposes on boiling, nitric and acetic acids being disengaged. A similar combination exists between the acetate and ferric chloride.

*Chromium acetates.*—Acetic acid enters into combination with chromous ( $\text{CrO}$ ), as well as with chromic oxide ( $\text{Cr}_2\text{O}_3$ ). The salts are not used in the industries and are only of scientific interest.

*Chromous acetate*,  $(\text{C}_2\text{H}_3\text{O}_2)_2\text{Cr} + \text{H}_2\text{O}$ , is prepared by mixing a solution of chromous chloride with sodium acetate. The salt separates out in small, lustrous red crystals which are sparingly soluble in water and alcohol, and quickly oxidize to a greater degree on exposure to the air, the succeeding salt being formed.

*Chromic acetate.*—A neutral salt is known and there are very likely several basic ones. The solution of the neutral salt, which is obtained by dissolving chromic hydrate in heated acetic acid, forms a red fluid, green in a reflected, and red in a transmitted, light. It is not decomposed by boiling, but by ammonia. The precipitate, however, redissolves, on adding ammonia in excess, to a violet-red fluid because the hydrate is soluble in ammonium acetate. Hence, a solution of the salt acidulated with acetic acid is not precipitated by ammonia.

There are also known crystallized combinations of this salt with chromic chloride and sulphate and nitrate of chromium.

If the solution of the neutral salt is for some time digested with chromic hydrate, it acquires a darker color, the acid reaction disappears, and on evaporating, a green powder soluble in water remains behind. Ordway has described a purple basic salt.

*Nickel acetate* forms small green crystals soluble in water, but not in alcohol.

*Cobalt acetate* forms small red crystals, the concentrated solution of which turns blue on heating but again red on cooling, and can, therefore, be used as sympathetic ink.

*Zinc acetate*,  $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2$ .—This salt may be prepared by dissolving metallic zinc, zinc oxide or zinc carbonate in acetic acid, or by the decomposition of zinc sulphate by acetates of lime or lead similar to the acetate of manganese. The acetate is in the first three instances simply obtained by evaporation, and in the latter, after agitating the mixture, filtering and evaporating the filtrate. The salt crystallizes in flexible, opalescent, six-sided tables which effloresce slightly in the air. Technically the best receipt is to dissolve 4 parts of the sulphate of zinc and  $7\frac{1}{2}$  parts of acetate of lead each in 3 parts of hot water, mixing the solutions, agitating, and after the sulphate of lead has deposited, drawing the clear liquid off to crystallize.

*Copper acetates. Cuprous acetate*,  $\text{Cu}_2(\text{C}_2\text{H}_3\text{O}_2)_2$ .—This salt is produced by subjecting crystallized verdigris to dry distillation. It is a white substance crystallizing in fine needles, which are decomposed by water into yellow cuprous hydrate and cupric acetate.

With cupric oxide acetic acid forms a normal and several basic salts.

*Neutral cupric acetate; crystallized verdigris*,  $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2$ .—The normal cupric acetate may be prepared by dissolving pure cupric oxide or cupric hydrate in pure acetic acid or by employing, instead of the pure oxide, copper scales whose content of metallic copper and of cuprous oxide is converted into cupric oxide by moistening with nitric acid and gentle glowing. The cupric oxide thus obtained is washed to remove foreign substances. The conversion of the cuprous oxide into cupric oxide is especially essential when the acetic acid is not entirely free from hydrochloric acid, as otherwise cuprous chloride is formed which dissolves with difficulty.

If copper scales cannot be obtained, hydrated basic carbonate of copper can be prepared by precipitating sulphate of copper with soda, and, after washing and pressing, dissolving in acetic acid. Sulphate of soda remains dissolved in the water, and this solution can eventually be utilized for the

conversion of crude calcium acetate into sodium salt. Instead of soda, milk of lime can also be used for the decomposition of the sulphate of copper, a mixture of calcium sulphate and cupric hydrate being precipitated. By adding acetic acid the latter is redissolved while the calcium sulphate remains suspended. When the latter has settled the solution is drawn off and evaporated. The calcium sulphate is repeatedly washed with small portions of water, and the wash-waters are used for dissolving fresh quantities of sulphate of copper.

In case the sulphate of copper contains iron, the latter is removed by digesting the solution for several days with basic carbonate of copper. The presence of iron is recognized by the sulphate not dissolving entirely in ammonia in excess, but leaving behind a red-brown residue (ferric hydrate).

The neutral acetate can also be prepared by dissolving the basic salt, verdigris (described below), in acetic acid. The solution is filtered and evaporated until a crystalline film is formed. This method is, however, expensive.

The method by double decomposition may be recommended for preparing the neutral acetate on a small scale, but not for manufacturing purposes. Sulphate of copper (125 parts) and sodium acetate (136 parts) decompose each other, neutral cupric acetate crystalizing out, while sodium sulphate remains in solution. The yield is, however, somewhat smaller than theoretically might be expected, because the sulphate of copper is not entirely insoluble in sodium sulphate solution. By this process the object is quickly accomplished, and for this reason is decidedly to be preferred to the following: Sulphate of copper (125 parts) and normal lead acetate (190 parts) decompose completely only in dilute, but not in concentrated, solutions. Hence strong evaporation is required, whereby acetic acid is lost. Further, with the use of lead acetate some of the newly-formed lead sulphate is obtained in solution; but the lead cannot be separated with sulphuretted hydrogen because the latter would also decompose the copper salt. The disadvantage of substituting calcium acetate for the lead ace-



tate is that it is not crystallized and hence furnishes no external criterion of purity ; in fact it always varies slightly in composition. If a small excess of calcium salt has been used, the latter, after the calcium sulphate is filtered off and the solution evaporated, does not remain in the mother lye, but crystallizes out as double salt (see below), together with the copper salt. Since these acetates create difficulties, and as each of them must first be prepared by the manufacturer by means of acetic acid, it would seem more rational to directly use this acetic acid for dissolving the cupric oxide, whereby no by-products of little value, such as sulphate of lead, calcium and sodium, are formed.

The evaporation of the solution of cupric acetate obtained by any of the above methods is effected in a copper boiler over an open fire, or, still better, by steam. It is recommended to close the boiler so that the escaping vapors of water and acetic acid are condensed in a worm. Independently of the fact that by these means the escaping acetic acid is regained and can be used for other purposes, a great advantage is that the air of the workroom is thereby not contaminated by flying particles of salt.

Crystallization is generally effected in stoneware pots into which dip a number of slender wooden rods. The pots are placed in a warm room. Crystallization is finished in about 14 days. The crystals turn out especially beautiful when the acid somewhat preponderates and the solution is cooled very slowly.

The salt forms dark green \* rhombic prisms of a nauseous metallic taste, which dissolve in 14 parts of cold, and 5 parts of boiling, water, and are also soluble in alcohol. Heated in the air the crystals burn with a green flame.

Neutral cupric acetate contains in 100 parts: Cupric oxide 39.8, anhydrous acetic acid 51.1, water 9.

On heating, the dilute solution of the neutral salt yields

\* There is also another salt of a beautiful blue color, which contains, however, 5 equivalents of water (Wöhler). It is prepared by exposing a solution of the salt mixed with free acetic acid to a low temperature. At 95° F. it passes into the ordinary green salt.

acetic acid and deposits a basic salt ; hence the use of strongly diluted acetic acid or even distilled vinegar is not suitable for the preparation of crystallized verdigris. By long-continued digestion with freshly glowed charcoal the dilute solution yields its entire content of copper to the latter ; hence vinegar containing copper can be purified in this manner (2 or 3 per cent. of charcoal being sufficient). The crystals of normal cupric acetate, after drying *in vacuo*, lose more water at 212° F., but give off 9 per cent. of their water between 230° and 284° F. By destructive distillation cupric acetate yields strong acetic acid which contains acetone and is contaminated with copper. Cuprous oxide ( $\text{Cu}_2\text{O}$ ) is obtained in red octahedral crystals when the neutral salt is heated with organic substances, such as sugar, honey, starch, etc. With the acetates of potassium, sodium, and calcium, normal cupric acetate gives double salts of a vivid blue color, which form fine crystals.

The chief use of normal cupric acetate in the arts is in making pigments and for resisting the blue color which the indigo would communicate in the indigo bath of the calico printer. In the latter case its mode of action depends on the readiness with which it parts with oxygen, whereby the indigo is oxidized before it can exert any action on the cloth, being itself reduced to the state of acetate of suboxide of copper. Crystallized verdigris is occasionally employed as a transparent green water color or wash for tinting maps. In medicine it is used for external application. It is poisonous, like all soluble copper salts.

*Basic cupric acetates.* *Sesquibasic cupric acetate* ( $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2$ )- $\text{CuO}+6\text{H}_2\text{O}$ .—This compound is obtained pure by gradually adding ammonia to a boiling concentrated solution of the normal acetate until the precipitate, which is at first formed, is redissolved. As the liquor cools the new salt then crystallizes out in beautiful blue-green scales, which at 212° F. lose 10.8 per cent. of their water. Their aqueous solution is decomposed by boiling, acetic acid being given off and the black oxide of copper precipitated.

*Dibasic cupric acetate*,  $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2\text{CuO} + 6\text{H}_2\text{O}$ , constitutes the greater part of the blue variety of verdigris. It forms beautiful, delicate, blue, crystalline needles and scales, which when ground form a fine blue powder. When heated to  $140^\circ$  F. they lose 23.45 per cent. of water and become transformed into a beautiful green, a mixture composed of the neutral and tribasic acetates. By repeated exhaustion with water the dibasic, is resolved into the insoluble tribasic, salt, and a solution of the normal and sesquibasic cupric acetates.

*Tribasic cupric acetate*,  $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2\text{CuO} + 3\text{H}_2\text{O}$ .—This compound is the most stable of all of the acetates of copper. It is prepared by boiling the aqueous solution of the neutral acetate, by heating it with alcohol, by digesting its aqueous solution with cupric hydrate, or by exhausting blue verdigris with water as above mentioned. The first methods yield the salt in the form of a bluish powder composed of needles and scales, the last as a bright green powder. This salt yields all its water at  $352^\circ$  F. ; at a higher temperature it decomposes and evolves acetic acid. Boiling water decomposes the solid tribasic acetate into a brown mixture of the same salt with cupric oxide.

Under the name of *verdigris* two varieties of basic cupric acetates are found in commerce: *French verdigris* which occurs in globular, bluish-green, crystalline masses, but also in amorphous masses, and *English verdigris* of a pure green color and crystalline structure, which is, however, also manufactured in Germany and Sweden.

The first variety is chiefly manufactured in the region around Montpellier, France. The refuse of grapes, after the extraction of the juice, is placed in casks until acetous fermentation takes place. The casks or vessels are covered with matting to protect them from dirt. At the end of two or three days the fermenting materials are removed to other vessels in order to check the process, to prevent putrefaction. The limit to which fermentation should be carried is known by introducing a test-sheet of copper into the mass for 24 hours; if, on withdrawing



it at the end of that time, it is found covered with a uniform green coating, the proper degree of fermentation has been reached.

Sheets of copper are prepared by hammering bars of the metal to the thickness of about  $\frac{1}{24}$  of an inch (the more compact the copper sheets the better), and they are then cut into pieces of 6 or 8 inches long by 3 to 4 broad. Sometimes old ship-sheathing is used and cut into pieces of the required size. The sheets are immersed in a concentrated solution of verdigris and allowed to dry. When the materials are all found to be in proper condition, the copper sheets are laid on a horizontal wooden grating in the middle of a vat, on the bottom of which is placed a pan of burning charcoal, which heats them to about 200° F. In this state they are put into large stoneware jars with alternate layers of the fermenting grape lees; the vessels are covered with straw mats and left at rest. At the end of 10 to 20 days they are opened to ascertain if the operation is complete. If the upper layer of the lees appears whitish and the whole has worked favorably, the sheets will be covered with silky crystals of a green color. The sheets are then taken from the jars and placed upright in a cellar, one against the other. At the end of two or three days they are moistened with water and again placed to dry. The moistening with water is continued at regular intervals of a week for six or eight times. This treatment causes the sheets to swell and become incrustated with increased coatings of the copper salt, which are detached from the remainder of the sheets by a copper knife. The scraped plates are submitted to a fresh treatment till the whole of the copper is converted into verdigris. The salt scraped off is made into a consistent paste by kneading with a little water, and in this state is packed into leathern bags which are placed in the sun to dry until the mass hardens and forms the tough substance which constitutes the commercial article.

In England, Germany and Sweden copper sheets are moistened with a solution of verdigris in vinegar and placed in a warm room, or woollen cloths moistened with the above solu-

tion are used, which are placed alternately with the copper sheets in a square wooden box. The woollen cloths are moistened with the solution every three days for 12 or 15 days, when small crystals commence to form on the sheets. The sheets are then drawn every six days through water and replaced in the box, but not in direct contact with the woollen cloths, small disks of copper or small pieces of wood being placed between each cloth and sheet. The woollen cloths are now more thoroughly saturated than before, but with a weaker solution. With a temperature of from  $54^{\circ}$  to  $59^{\circ}$  F., 6 to 8 weeks are required before the verdigris can be scraped off. The product is not identical with that obtained by the French method, it being somewhat poorer in acetic acid, and hence its color is not bluish-green, but almost pure green.

*Lead Acetates.*—With plumbic oxide acetic acid gives a neutral, as well as several basic, salts. The most important of these combinations are the neutral salt, known in commerce as sugar of lead, and a basic salt by means of which white lead is obtained.

*Neutral Acetate of Lead (Sugar of Lead),*  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 + 3\text{HO}$ .—According to Völkel's method, acetic acid prepared from wood-vinegar and rectified over potassium dichromate is saturated with litharge, filtered or decanted, and after a further addition of acetic acid until a slightly acid reaction takes place, evaporated to the crystallizing point.

By saturating acetic acid with litharge, a solution of basic salt is obtained, which is later on converted into neutral salt by the addition of acetic acid. This is more suitable than using only as much litharge as the acetic acid requires for the formation of the neutral salt, because the litharge dissolves with greater ease in solution of sugar of lead than in acetic acid.

Solution of sugar of lead, like solution of neutral cupric acetate, permits of the evaporation of acetic acid in boiling; and, hence, it is best to use strong acetic acid, because less will have to be evaporated and the loss of acetic acid be consequently

smaller. By taking, for instance, acetic acid of 1.057 specific gravity, for 100 lbs. of it 82 lbs. of litharge are required for the formation of the neutral salt. A larger quantity is, however, taken (from 100 to 180 lbs.), so that a basic salt is formed, or, with 100 lbs., a mixture of neutral and basic salts. To recognize the point of neutralization in the subsequent addition of acetic acid, litmus paper is used, or, still better, dilute solution of corrosive sublimate (1 part of corrosive sublimate in 100 of water), which does not change the neutral salt, but produces turbidity in the basic (Büchner). Hence, by from time to time testing the lead solution with this reagent, the point of neutralization is reached the moment turbidity ceases. This test is better than with litmus, considerable experience being required to hit the right point with the latter on account of solution of sugar of lead showing a slight, but perceptible acid reaction.

The solution of litharge in acetic acid is promoted by heat, and is effected either in a copper pan, the bottom and sides of which are brought in contact with a few bright sheets of lead (to prevent the copper from being attacked), or in a lead pan over an open fire, or in a wooden vat into which steam is introduced. The clear solution is evaporated. If this is to be done over an open fire, it is recommended to have a preparatory heating pan for each evaporating pan, as described in the preparation of calcium acetate, the preparatory heating pan, which is heated by the escaping gases, being used for the solution of the litharge in acetic acid. Lead pans, if used, should rest upon strong cast-iron plates. The dimensions of the pans vary very much. According to Assmus, they are  $6\frac{1}{2}$  feet long, 4 feet wide, and from 12 to 14 inches deep, while the depth of the preparatory heating pans is from 24 to 28 inches. From the latter, which stand at a higher level, the clear solution is discharged, through a stop-cock just above the bottom, into the evaporating pans. Evaporation should be effected at a moderate heat; actual boiling must be strictly avoided, as otherwise large losses of acetic acid are unavoidable and the solution readily acquires a yellow coloration.



According to the degree of evaporation (to  $36^{\circ}$  B. or to  $46^{\circ}$  B. or more) of the sugar of lead solution, distinct crystals are obtained or only a radiated crystalline mass. With a perfectly pure solution the first method is the best, since crystals bring a better price. The mother-lye, after being again acetified, is once more evaporated and acetified and yields more crystals.

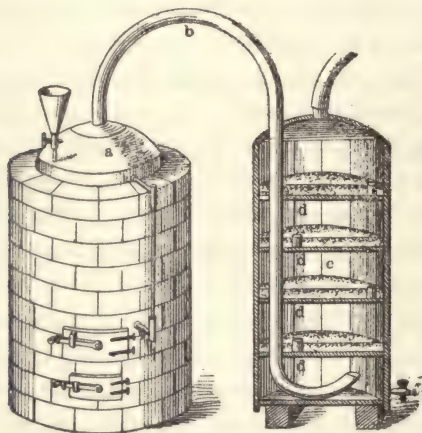
Stein recommends the conducting of the vapors of acetic acid or of vinegar into litharge mixed with a very small quantity of water. This method is in general use in Germany. But as the extract remaining in the still retains a considerable quantity of acetic acid, especially if beer had been added to the liquid used in the preparation of the vinegar, it is advisable to increase the boiling point of the latter by the addition of one-third of its weight of common or rock salt. At first the water condenses in the receiver and the volume of the fluid containing the litharge increases, but when the boiling point is reached in the condensing vessels, only the acetic acid is retained, while the litharge is first converted into sexbasic and then into tri-basic acetate. To obtain neutral salt, however, either the vapors must be somewhat expanded or several condensing vessels be placed one after the other.

Fig. 84 shows the distilling apparatus, consisting of a still, *a*, of sheet-copper. The vapors pass through a copper pipe, *b*, into the wooden vat, *c*, lined with lead, and about 35 inches in diameter and 67 inches deep. In this vat are four bottoms, *d*, of thick lead provided with fine perforations. Short lead pipes, soldered into these bottoms and arranged as shown in the figure, serve to conduct the vinegar vapors in the vat to and fro in the interspaces between the lead bottoms. For each still at least three of such vats are connected with each other. Upon the lead bottoms is first placed a layer of linen or of flannel, and next a layer of litharge 2 to 4 inches deep. To prevent the litharge from packing, it is mixed with an equal volume of pebbles, about the size of a pea. The vats are provided with lids of sheet-copper lined with lead. From the lid of the last vat a pipe leads to a worm surrounded with cold

water. The stop-cocks on the bottoms of the vats permit the discharge of the collected lead solution, which is effected (with the use of acetic acid) when it shows a specific gravity of at least  $36^{\circ}$  Bé. The solution being, however, basic, it is acetified with strong acetic acid, and brought into the crystallizing vessels.

This method is decidedly the best, because the evaporation of the solution is entirely or almost entirely omitted and the air of the workroom is not contaminated by particles of sugar of lead, which is very injurious to the health of the workmen.

FIG. 84.



Furthermore, this method does not require the use of pure acetic acid, since the impurities remain in the still. This, however, holds good only for non-volatile impurities. For the production of colorless salt, the crude acetic acid from wood-vinegar must necessarily be purified, as above mentioned, by potassium chromate and sulphuric acid.

The crystallizing pans are either of stone-ware or of wood lined with lead or thin copper, to which is soldered a strip of lead down the sides and across the bottom, with the idea of rendering the metal more electro-negative, so as to prevent the

acetic acid from acting on it. The wooden crystallizing pans are about 4 feet long by 2 feet wide, and from 6 to 8 inches deep, sloping inwards at the edges. Shallow, slightly conical copper vessels, 6 inches deep, with a diameter of  $29\frac{1}{2}$  inches at the bottom and  $31\frac{1}{2}$  inches at the top, are also used. The stone-ware pans are placed upon a slightly inclined level covered with lead. In these small pans crystallization is complete in 24 hours, while from 48 to 72 hours are required with the use of the larger wooden vessels. Crystallization being complete, the mother-lye is removed, and the vessels are placed upon a wooden frame over a gutter of sheet-lead to drain off, as shown in Figs. 85 and 86.

FIG. 85.

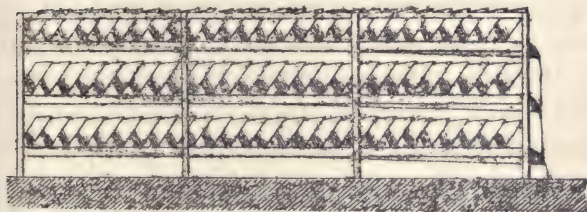


FIG. 86.



If especially beautiful crystals are to be obtained, the first crystals, which are not very distinct, are again dissolved in the water obtained by the condensation of the vapors escaping from the still. The solution being evaporated to the proper density is again allowed to crystallize. The crystals, after sufficient drainage, are placed upon linen spread over wooden hurdles and dried at a moderate heat, not exceeding  $75^{\circ}$  F. In some factories the heated air of a stove, placed outside the drying-house, is conveyed through pipes passing round the interior; at other places steam heat is employed for this purpose, which is much to be preferred, on account of its being more easily regulated.

When working on a large scale a centrifugal is advantageously employed for the separation of the mother-lye, in the



same manner as recommended for the preparation of sodium acetate.

Litharge being a quite impure lead oxide never dissolves entirely, and frequently contains over 10 per cent. of impurities, consisting of sand, clay, red lead or minium ( $\text{Pb}_3\text{O}_4$ ), metallic lead, traces of silver, cupric and ferric oxides. The cupric oxide passes into the sugar of lead solution and colors it slightly blue. To separate the copper, bright sheets of lead are dipped into the solution, the copper separating upon them in the form of a dark slime. The sheets of lead must be frequently cleansed (scraped), as otherwise they lose their effect. When there is a large accumulation of litharge residue, it can be worked for silver.

Sugar of lead can also be prepared from metallic lead, the process having been recommended first by Berard, and is said by Runge to yield a good product with great economy. Granulated lead, the tailings in the white lead manufacture, etc., are put in several vessels, say eight, one above the other, upon steps, so that the liquid may be run from one to the other. The upper one is filled with acetic acid, and after half an hour let off into the second, after another half hour into the third, and so on to the last or eighth vessel. The acid causes the lead to absorb oxygen so rapidly from the air as to become hot. When the acid runs off from the lowest, it is thrown on the uppermost, vessel a second time and carries off the acetate of lead formed. After passing through the whole series the solution is so strong that it may be evaporated at once so as to crystallize.

Apparently this method has a considerable advantage over that with litharge, metallic lead being cheaper and producing more sugar of lead (entirely free from copper) than litharge, because 103.5 lbs. of pure lead yield 189.5 lbs. of sugar of lead, while the same quantity is only obtained from 111.5 lbs. of pure litharge. Furthermore, commercial lead is always purer than litharge. On the other hand, this process has the disadvantage of a considerable quantity of acetic acid being lost by

evaporation on account of it having to pass through several vessels. The manufacture of sugar of lead is most suitably combined with that of white lead, it being thus possible to utilize the tailings, etc., to greater advantage than, as is frequently done, by melting them together and remelting, which always causes considerable loss.

Sugar of lead is further formed by boiling lead sulphate with a very concentrated solution of barium acetate, barium sulphate (permanent white) being thereby precipitated. For 100 parts of lead sulphate 84 parts of anhydrous or 100 of crystallized barium acetate are required, the yield being 125 parts of sugar of lead. Sulphate of lead is obtained in large quantities as a by-product in the preparation of aluminium acetate.

For many purposes of dyeing and printing the use of pure sugar of lead is not necessary, the *brown* acetate of lead answering all requirements. For its preparation ground litharge is introduced in small portions, stirring constantly, into distilled wood vinegar in a vat until red litmus paper is colored blue, and, hence, a basic salt is formed. The impurities separating on the surface are removed and the clear fluid is then transferred to a copper pan equipped with strips of lead, and is evaporated to about two-thirds its volume, the brown smeary substances rising to the surface during evaporation being constantly removed. By again diluting and slightly acidulating the concentrated fluid a further portion of the foreign substances can be removed. Finally evaporation is carried to the crystallizing point, *i. e.*, until a few drops congeal when allowed to fall upon a cold metal plate. The addition of animal charcoal for the purpose of discoloration is of no advantage. The coloration is not completely removed, and the little effect produced is attained at a considerable loss of salt, which is absorbed by the animal charcoal.

By disturbing crystallization by constant stirring during cooling, a nearly amorphous mass, having the appearance of yellow wax, is obtained, which is much liked by many con-

sumers. The product thus obtained is not always a neutral salt, but sometimes a mixture of neutral and basic salts (besides empyreumatic substances). After cooling it must, therefore, be quickly and well packed, in order to protect it from the moisture and the carbonic acid of the air. The sugar of lead solution may, however, also be evaporated only so far that some mother-lye remains after cooling, the crystallized mass being then for some time allowed to stand in a moderately warm room. In consequence of capillarity, the impurities, which occur chiefly in the mother-lye, gradually rise up between the crystals, a slight coating of yellow, or brown, smeary substance being finally formed upon the mass of crystals, which can be readily removed.

The linen upon which the crystals are dried must be carefully protected from fire, as it ignites from the slightest spark and burns like tinder.

If the hot solution be set aside to cool rapidly, the sugar of lead crystallizes in clusters of fine needles; but if evaporation be conducted slowly the crystals are truncated and flattened, quadrangular and hexahedral prisms derived from a right rhombic prism. Acetate of lead has a sweet astringent taste, is soluble in  $1\frac{1}{2}$  parts of water and in 8 parts of ordinary alcohol. The crystals are permanent in the atmosphere, but are apt to effloresce and become anhydrous if the temperature ranges between  $70^{\circ}$  and  $100^{\circ}$  F.

Acetate of lead consists of:

Plumbic oxide . . . . .	58.9
Anhydrous acetic acid . . . . .	26.9
Water . . . . .	14.2
	<hr/>
	100.00

Aqueous solution of sugar of lead slightly reddens litmus-paper, but shows an alkaline reaction upon turmeric, browning it.

At  $167^{\circ}$  F. the crystals of acetate of lead melt, and slowly yield up their water; by heating the entirely dephlegmated



salt more strongly it fuses at  $536^{\circ}$  F. to a clear, oil-like, colorless fluid and decomposes above this temperature, evolving all the compounds usually obtained in the destructive distillation of the acetates of the heavy metals, while a residue of metallic lead in a very minute state of division, with some charcoal, is left behind. When this distillation is conducted in a glass tube closed at one end and having the other drawn out for convenience of sealing at the end of the operation, the well-known lead pyrophorous is made. The particles of metallic lead are so small that, when thrown into the air, oxygen molecules come into such intimate contact with them that ignition is effected from the rapidity with which lead oxide is formed.

A slight decomposition occurs when the neutral salt is exposed to an atmosphere of carbonic acid, carbonate of lead being formed; the portion of acetic acid thus liberated protects the remainder from further change.

Cold solution of sugar of lead is not immediately changed by ammonia; by adding, however, a large excess of it, sesquiacetate of lead is gradually separated; on boiling, yellow-red crystalline lead oxide is precipitated.

The introduction of chlorine gas into a solution of sugar of lead produces in a short time a brown precipitate of plumbic dioxide. Bromine acts in a similar manner, but on account of its insolubility, iodine produces scarcely any effect.

Solution of calcium chloride at once produces a yellow precipitate, which gradually becomes brown.

Sugar of lead containing considerable copper has a bluish appearance. If the content of copper is small, it is recognized by the solution acquiring with ammonia a blue coloration, or, still better, by mixing the solution of sugar of lead with an excess of solution of Glauber's salt and testing the filtrate with potassium ferrocyanide. A dark-red precipitate indicates copper.

Sugar of lead, as well as the basic lead salts to be mentioned further on, possesses poisonous properties.

Sugar of lead is chiefly used for the preparation of alum-

inium acetate, as well as of other acetates. Considerable quantities of it are consumed in the manufacture of colors, for instance, of neutral and basic lead chromate, chrome yellow, chrome orange, and chrome red. Upon the cloth-fibre (especially wool) chrome yellow and chrome orange are produced by means of sugar of lead, particularly with the brown variety; the latter product being also very suitable for the production of the so-called chrome green, which is obtained by the joint precipitation of chrome yellow and Berlin blue.

Neutral lead acetate gives crystallizable double salts with potassium acetate and sodium acetate as well as with lead nitrate, lead chloride, lead bromide, etc.

*Basic lead acetates.*—Several of these compounds are known. Those with 2 and 3 equivalents of lead oxide to 1 equivalent of acetic acid are soluble in water, show a strong alkaline reaction, and with carbonic acid the solutions yield at once and in every degree of concentration, abundant precipitates of white lead (basic carbonate of lead), while, when the operation is at a suitable moment interrupted, neutral salt remains in solution. In this manner white lead is manufactured according to the so-called French method (of Thénard and Roard) at Clichy and other places in France, as well as in different German factories. If however, the introduction of carbonic acid be continued until no more precipitate is formed, a part of the lead of the neutral salt is also precipitated as carbonate, which, however, is neutral, and an acid solution remains behind.

The soluble salt known as *lead vinegar* or *extract of lead* is prepared by digesting 2 parts of sugar of lead dissolved in 5 of water with 1 of finely powdered litharge. The proportional quantities of sugar of lead, litharge and water prescribed by the Pharmacopœias of the different countries vary very much, and, consequently, also, the compositions and specific gravities (from 1.20 to 1.36) of the solutions of lead prepared in accordance with them. The litharge dissolves very readily in the sugar of lead solution, in fact with greater ease than in

acetic acid, and especially with greater rapidity if the sugar of lead solution be heated in a silver dish to the boiling point and the litharge gradually introduced. For the manufacture on a large scale, the sugar of lead solution and the litharge may be brought into a barrel revolving around its axis. If the operation is to be conducted at the ordinary temperature, the barrel must be closed to prevent the access of the carbonic acid of the air. Very remarkable is the behavior of the tribasic acetate towards hydrogen dioxide; plumbic dioxide being first formed. But in a short time this exerts a decomposing influence upon the hydrogen dioxide which may be present in excess, so that both dioxides now lose one-half of their oxygen, which evolves in the form of gas, and water and plumbic oxide are formed.\* Now, as freshly precipitated plumbic dioxide possesses the further property of decomposing solution of potassium iodide, Schoenbein recommends tribasic acetate of lead, together with paper coated with paste prepared with potassium iodide, as the most sensitive reagent for hydrogen dioxide.

*Lead Sesquibasic Acetate, Triplumbic Tetracetate.*—This salt is obtained by heating the diacetate until it becomes a white, porous mass; this is redissolved in water and set aside to crystallize. Sesquibasic acetate is soluble in both water and alcohol; its solutions are alkaline.

*Tribasic acetate of lead* is prepared by digesting 189.5 lbs. of sugar of lead with 223 lbs. of plumbic oxide (pure) or 3 lbs. of sugar of lead to 4 lbs. of litharge; or, according to Payen, into 100 volumes of boiling water are poured 100 volumes of aqueous solution of sugar of lead saturated at 86° F., and afterwards a mixture of pure water at 140° F., with 20 volumes of ammonia liquor free from carbonate. The vessel is then immediately closed, and in a short time an abundance of the tribasic acetate crystallizes out. The salt presents itself under the form of long needles. It is insoluble in alcohol, very solu-

\* Schoenbein in Wagner's Jahresbericht, 1862.



ble in water, its solution being alkaline. Tribasic acetate is the most stable of all the subacetates of lead. It takes a leading part in the manufacture of white lead by the Clichy process. It is, in point of fact, a solution of this salt, which is decomposed by the carbonic acid, and gives rise to the carbonate of lead, being itself at the same time converted into lead diacetate. In the Dutch process the formation of lead carbonate is, according to Pelouze, also due to the formation of tribasic acetate on the surface of the sheets of lead, which is, in its turn, decomposed by the carbonic acid.

*Sexbasic Acetate of Lead.*—This body is prepared by digesting any of the preceding salts with lead oxide. It is a white powder slightly soluble in boiling water, from which it crystallizes out in silky needles which consist of two equivalents of the salt combined with three equivalents of water.

*Uranium Acetate.*—With uranous oxide, acetic acid combines to a dark green crystallizable salt, and with uranic oxide to a yellow basic salt, which, combined with water, appears in two different forms of crystals. It is remarkable for giving, with many other acetates, well crystallizing salts, of a beautiful color, and partly showing magnificent dichroism (Wertheim and Weselsky).

*Tin acetate* is prepared by dissolving stannous hydrate\* in heated strong acetic acid, or by mixing stannous chloride ( $\text{SnCl}_2$ ) with acetate of sodium or calcium. It forms small colorless needles, which have a strong metallic taste and readily decompose in the air. The salt is used to discharge azo-dyestuffs in calico printing.

*Bismuth Acetate.*—Bismuth nitrate prepared by gradually introducing pulverized metallic bismuth into cold dilute nitric acid is mixed with pure concentrated sugar of lead solution. The salt separates in small, colorless needles.

*Mercurous acetate* can be prepared by dissolving pure mer-

\* The hydrate is obtained by precipitating stannous chloride with soda lye and washing the precipitate.

curous oxide or its carbonate in acetic acid, or by mingling hot solutions of mercurous nitrate and acetate of sodium or of potassium. The pure mercurous carbonate is heated to boiling with 8 parts of water, and concentrated acetic acid added until all is dissolved; the hot, filtered liquid free from oxide being allowed to cool. Or, acidulated nitrate is diluted with 6 to 8 parts of water, heated and mixed with one equivalent of acetate of sodium or potassium, dissolved in 8 parts hot water containing a little free acid, and cooled. The salt, when separated, is washed with a little cold water, dried in the dark at a gentle heat, and kept from the light in covered bottles.

It crystallizes in fine, white, silvery scales, flexible and unctuous to the touch, with a nauseous metallic taste, easily decomposed by light. It dissolves with difficulty in cold water, requiring 33 parts at the ordinary temperature. It is partially decomposed by boiling water into acid and basic salts of both oxides and metallic mercury. It is used in pharmacy.

*Mercuric Acetate.*—Dissolve red oxide of mercury in concentrated acetic acid at a gentle heat and evaporate to dryness, or partially to crystallization, or by spontaneous evaporation. When obtained by the first process it is a white saline mass; by the second it forms crystalline scales; and by the third, four-sided plates, which are partly transparent, partly pearly and translucent, anhydrous, of a nauseous metallic taste, fusible without decomposition, solidifying to a granular mass, but the point of decomposition of the latter is near that of fusion. It dissolves in 4 parts of water at 50° F., in 2.75 at 66.2° F., and in 1 at 212°, but by boiling it is partly decomposed, red oxide being separated. Even in the air its solution suffers the latter change and contains a basic salt. With free acetic acid it is not decomposed. One hundred parts of alcohol dissolve  $5\frac{2}{3}$  of this salt, and this solution behaves like the aqueous one. It generally contains, except when carefully crystallized, some mercurous oxide.

*Silver Acetate.*—This salt is obtained by precipitating a concentrated solution of silver nitrate with a concentrated solution

of sodium acetate. It forms a white crystalline precipitate. It dissolves in about 100 parts of cold, but readily in hot, water, and only sparingly in alcohol. On exposure to light it acquires a dark color, being partially reduced. On heating, it yields acetic acid, metallic silver remaining behind.

If the salt be heated with disulphide of carbon in a closed glass tube to 329° F., silver sulphide, carbonic acid and anhydrous acetic acid are formed (Broughton).

On treating the dry salt with iodine, lively decomposition takes place, whereby silver iodide, some metallic silver and coal remain behind, while methyl oxide, acetic acid, acetylene and hydrogen appear. With iodine a solution of this salt yields acetic acid, silver iodide and iodate of silver (Birnbbaum).

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## CHAPTER XXVI.

### PREPARATION OF PURE WOOD SPIRIT OR METHYL ALCOHOL, AND OF ACETONE, AND WORKING THE WOOD TAR.

*Preparation of Wood Spirit.*—The crude wood-spirit solutions collected during the distillation of the wood vinegar contain, according to their method of production, from 9 to 10 per cent. wood spirit. They are subjected to repeated distillation and rectification over milk of lime to fix the acetic acid present and to saponify the methyl acetate. In this manner wood spirit is produced, *i. e.*, a mixture that besides methyl alcohol contains other combinations such as aldehyde, methyl-acetic ether, acetone and similar combinations, amines, higher alcohols, etc.

The crude wood-spirit of commerce contains besides the above-mentioned constituents, 75 per cent. of methyl alcohol. It is clear as water to dark brown and can be mixed in every proportion with water without becoming turbid.

Wood spirit for denaturing purposes is produced from the



crude wood spirit by further rectification. It contains 95 per cent. methyl alcohol, and while the higher alcohols and the acetone have been removed, aldehyde, methyl acetate, etc., are still present.

Pure wood spirit contains 98 to 99.5 per cent. wood-spirit constituents, among which methyl alcohol, however, preponderates so that they are almost entirely pure. Such pure wood spirit contains only very small quantities of acetone—0.01 to 0.5 per cent. It does not discolor bromide solution and when mixed with concentrated sulphuric acid acquires an only slightly yellower color.

The rectification of the crude wood-spirit solutions collected during the distillation of the wood vinegar is effected in a columnar still. The crude wood-spirit solution is brought into the still and, after adding slaked lime, the fluid is allowed to rest for several hours. After the addition of the lime the fluid soon becomes strongly heated in consequence of the free acids combining with the lime, and of the formation of calcium acetate and methyl alcohol from the methyl acetic ether, small quantities of ammonia being also evolved.

After having rested for several hours the fluid is subjected to distillation. In Fig. 87, *a* represents the copper still, *b* an ellipsoidal or egg-shaped vessel which serves as a receiver, and *c* the rectifying apparatus, consisting of a series of Pistorius's basins into the uppermost of which a moderate current of water is conducted; *d* is the condenser.

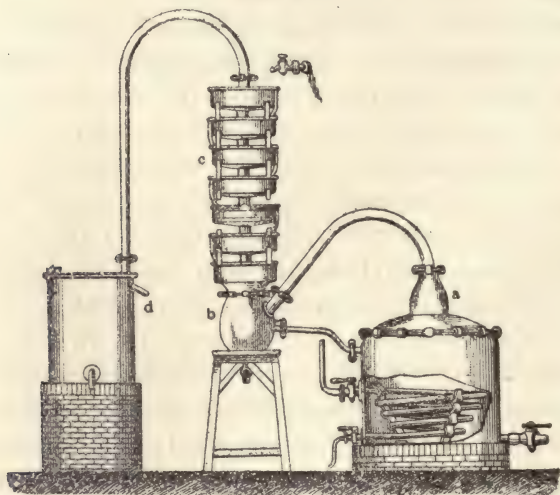
The still *a* has a capacity of 1000 to 1200 quarts; the steam pipe placed in it is 2 inches in diameter and 32 feet long. The vapors pass out through the wide pipe in the cover, and what is condensed in *b* runs back through a narrower pipe into *a*. In the rectifying vessel or rather dephlegmator, the rising vapors are forced to pass around a copper disk placed in each basin, and thus to come in contact with the surface of the basin cooled by water. From this it is evident that the less volatile bodies are condensed in the basins and run back into *b* and from there into *a*, while the more volatile vapors

pass through the swan-neck and are condensed in *d*. Much, of course, depends on the quantity (and temperature) of the water running into the rectifying vessel.

With a rectifying vessel consisting of seven basins, each 1.64 feet in diameter, and with a correctly conducted inflow of water, a product of 0.816 specific gravity is obtained by one operation from crude wood spirit of 0.965 specific gravity.

This product can be used for many purposes, for instance in the preparation of varnishes. It is, however, not entirely

FIG. 87.



pure, being rendered turbid by water which is due to a content of the previously mentioned hydrocarbons; it further contains some acetone, methyl acetate, aldehyde, ammonia, methylamine, and is not fit for use in the production of aniline colors.

For further purification, this rectified wood-spirit is diluted with water until it shows a specific gravity of 0.934, and is then allowed to rest a few days, when the greater portion of the hydrocarbons has separated as an oily layer on the top. The clear fluid is now again rectified with an addition of 2 to

3 per cent. of lime whereby a distillate is obtained which does not become turbid with water, but in time turns yellow.

For the preparation of wood spirit suitable for denaturing purposes, the crude wood spirit is diluted with water to from 30 to 40 per cent., compounded with milk of lime—20.30 liters to every 1000 liters of spirit—and carefully and slowly rectified from large columnar stills for several days, whereby the following fractions are obtained :

1. First runnings containing acetone, with 60 to 80 per cent. acetone. 2. High per cent. intermediate runnings, giving bright mixtures with water, and containing 7 to 10 per cent. of acetone. 3. High per cent. intermediate running, not giving bright mixtures with water. 4. Allyl alcohol-like after runnings below 90 per cent. 5. After runnings containing oil.

If the first of these fractions be diluted with water—100 liters of water to 200 kilogrammes of distillate—and acidulated with somewhat more sulphuric acid than required for fixing the bases and again distilled from an iron or copper still lined inside with lead, a product suitable for denaturing purposes is obtained. For this purpose all the fractions are used which are miscible with water without becoming turbid and are so rich in acetone that the mixture finally contains at least 30 per cent. of it and has a specific gravity of 90° Tralles.

For the further treatment of fraction 2, water in the proportion of 1:2 is also added and then 1 to 3 per cent. of soda lye. The object of the addition of soda lye is to fix the phenol-like body, to saponify the esters and resinify the aldehyde. During rectification the fractions that possess less than 0.1 per cent. acetone are caught by themselves and designated "pure methyl."

The third fraction is treated in the same manner but in place of soda lye, sulphuric acid is added. The fourth fraction is so far diluted with water that the dissolved oils are separated. The latter are removed and the residue, after adding sulphuric acid, is again rectified, products which may also



partially serve as wood spirit for denaturing purposes being thus obtained.

*Preparation of Acetone.*—Acetone is a clear, mobile, ethereal-smelling liquid, boiling at 134° F., and of specific gravity 0.797 at 59° F. It is prepared by heating calcium acetate in retorts which are connected with a cooling apparatus. The calcium acetate used for the purpose must be pure and should be brought into the retorts in a perfectly dry and pulverized state. It is then slowly heated until no more fluid runs off from the cooler. The residue in the retorts consists of calcium carbonate, and is again used for the preparation of calcium acetate. Since acetone boils at a very low temperature, provision must be made for abundant cooling and it is best to use ice water for feeding the cooling apparatus.

The decomposition of the calcium acetate to acetone and calcium carbonate proceeds according to the following equation:



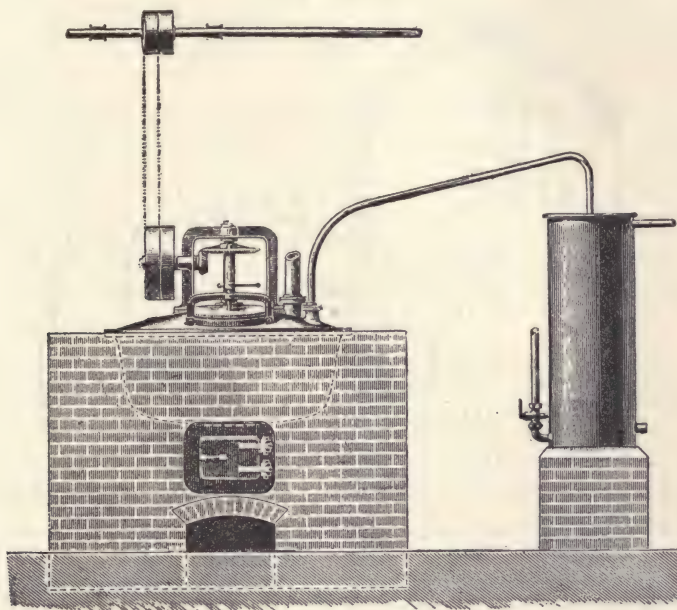
This decomposition commences already in a slight degree at 302° F., but takes place completely only at 752° F., and hence the use of uniform and very high temperatures is indispensable for the production of acetone. The theoretical yield from 200 lbs. of calcium acetate (gray acetate) amounts in round numbers to 66 lbs. of acetone. Since the gray acetate contains besides calcium acetate other combinations, for instance, calcium butyrate and propionate, the yield may be materially less, and may in round numbers be given as about 44 lbs. of acetone (dimethylketone). The homologues mentioned are of course also decomposed in a manner similar to the calcium acetate, but higher ketones are then formed which in the purification of the crude acetone, yield the so-called acetone oils.

The decomposition of the calcium acetate is as a rule effected in a cast-iron pan, Fig. 88, furnished with a powerful stirrer and a man-hole for charging the calcium acetate. The temperature for decomposition should not exceed 752° F., and is

controlled by a pyrometer. The vapors evolved pass first through a dust-separator and then through a pipe into a condenser, where they liquefy.

The crude distillate is rectified. This first distillate is diluted to one-half with  $\frac{1}{2}$  volume of water and again rectified, whereby a product with 90 per cent. acetone is obtained. The last distillation is effected with the addition of potassium permanganate. The first two or three liters of distillate are

FIG. 88.



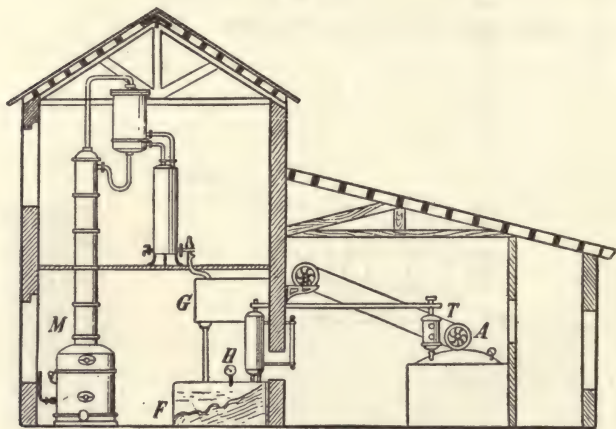
caught by themselves, and then all that boils between  $122^{\circ}$  and  $136.4^{\circ}$  F.

Fig. 89 shows the arrangement of a plant for the production of acetone. It contains several decomposing apparatuses for the production of crude acetone. The pipes conducting the vapors enter first a common collecting pipe, and from there are conducted to the condenser which terminates in the collecting vessel *F*, the quantity of fluid in it being indicated by

the float *H*. The decomposing apparatuses are separated by a wall from the condenser and rectifier; the fireplaces are outside the working room. The decomposing apparatus is equipped with a dust collector *T*, above which is a broad T-pipe for conducting the vapors. This pipe also serves for the purpose of cleaning the dust collector.

From the collecting vessel the crude acetone is pumped into the rectifier *M*, the latter being similar in arrangement to an

FIG. 89.



apparatus for rectifying alcohol. The pure acetone is caught by itself in the vessel *G*.

The use of acetate of barium, strontium or magnesium in place of calcium acetate is more advantageous. The drawback with the use of calcium acetate consists in that many tarry substances pass over and clog the pipes, and, besides, the distillate is contaminated with empyreumatic substances.

The preparation of pure acetone is not very easy, notwithstanding its apparent simplicity, and requires the use of perfectly separating columnar stills and experience, for the reason that the admixtures of the acetone have nearly the same boiling points. The acetone vapors are inflammable and when mixed with air explosive.



According to F. H. Meyer's system, German patent 134,978, pure acetone is manufactured by spreading the gray acetate in layers 2 to 4 centimeters deep upon sheets or sieves, which rest upon trucks and are pushed into the distilling muffles. These muffles are capable of working up 4400 lbs. of calcium acetate in 24 hours. They are heated by a direct fire, a uniform distribution of the heat and the same heating at all points of the charge being secured by proper regulation of the fire. When the acetone has been distilled off and the last remnants blown out with steam, the truck is removed from the muffle and is replaced by another previously charged with calcium acetate.

The acetone oils which, as previously mentioned, are obtained in the purification of crude acetone are decomposed to two groups, namely to white acetone oil which comprises the fractions boiling at from 167° to 466° F., and to yellow acetone oil boiling between 466° and 502° F. These oils are used in the celluloid industry and as additions to wood alcohol intended for denaturing purposes.

*Working the wood tar.*—Wood tar contains a large quantity of combinations of which, however, only the mixture found in commerce under the name of creosote can be separated to advantage. By itself wood tar may be utilized as a preservative coating for wood, as well as for obtaining soot, and eventually as fuel in the destructive distillation of wood.

While beech tar, for instance, contains without doubt considerable paraffin, it cannot be produced on a large scale at a price to compete with that of the product obtained from crude petroleum. The tar obtained from resinous woods contains oil of turpentine and can be worked to greater advantage than that from hard wood.

*Preparation of creosote and tar oils.* The wood tar is subjected to distillation, this being best effected in a horizontal still of the shape of a steam boiler and so bricked-in as to be slightly inclined towards one side. On the lowest part of the still is a larger aperture which can be closed by a cover and clamp.

The object of this arrangement is to facilitate the quick removal of the pitch-like or asphalt-like mass which remains behind in the still after the volatile products have been distilled off, and which, if allowed to become cold, adheres so firmly to the sides of the still that it can be detached only with great difficulty.

All the tarry substances, which separate in distilling wood-spirit from wood vinegar, and in neutralizing with lime or soda, are combined with the tar taken from the condensing vessels, and the mass thus obtained is subjected to distillation. The latter might be conducted so that the distillates resulting at certain temperatures are caught by themselves and the distillate fractionated; but, as a rule, the distillates are only separated in such a manner that only the light oleaginous products up to specific gravity 0.980 are caught by themselves and worked further, separately from the heavy oils of upward of 1.010 specific gravity.

At the commencement of distillation crude wood-spirit first passes over, which is followed by quite a quantity of acetic acid (distilled wood-vinegar). Next the light, and later on the heavy, oils pass over, a pitch-like residue remaining in the still. By mixing this residue, while still in a liquid state, with dry hot sand, blocks may be shaped from the mass thus obtained, which may be used for paving, like asphalt blocks. Mixed with culm it yields a dough-like mass which may be utilized for the manufacture of briquettes. If the residue cannot be utilized in any other manner, it may be allowed to run upon iron plates, and when cold, is broken up into small pieces and used as fuel together with coal.

The quantities of the separate products of distillation depend on the nature of the wood from which the tar has been obtained and on the manner in which destructive distillation has been conducted. Hard woods give on an average a tar which by distillation yields, according to Vincent :

Watery distillate (wood spirit, acetic acid) . . .	10 to 20 per cent.
Oleaginous light distillate, sp. gr. 0.966 to 0.977 .	10 to 15 “
“ heavy “ “ 1.014 to 1.021 .	15 “
Pitch . . . . .	50 to 62 “

The distillates, according to their specific gravities, are caught separately in vats, a sample, for instance, 1 quart of the fresh distillate, being immediately taken for the purpose of accurately determining the quantity of soda required for neutralizing the total quantity of fluid. The quantity of concentrated soda solution necessary for neutralization is then added to the distillate, the whole thoroughly mixed, and the fluid allowed to repose until two sharply separated layers are formed, the upper one of which is of an oleaginous nature. The watery fluid is then allowed to run off and is brought into one of the vats in which crude wood-vinegar is caught. The oleaginous layer is worked further by distillation.

The oils remaining after neutralizing the light and heavy distillates are combined and subjected to careful rectification. The receiver is changed as soon as it is ascertained by the thermometer that the temperature has risen above 302° F., and is again changed when the temperature rises above 482° F. The hydrocarbons distilling over at below 302° and above 482° F. might be used as solvents and for illuminating purposes, but their preparation is not remunerative.

The distillate which has passed over between 302° F. and 482° F. contains phenol, cresol and phlorol, which together form wood-creosote. The distillate is intimately mixed with the assistance of a stirring apparatus with highly concentrated soda lye (36° Bé.), and the watery fluid is drawn off from the supernatant layer of oil, which is combined with the other hydrocarbons. The watery fluid is for some time boiled in an open pan to expel any hydrocarbons still present, and is then saturated with sulphuric acid and allowed to repose. The fluid of a penetrating odor separated thereby is creosote, which is used for medicinal purposes. As a disinfecting agent it has, however, been superseded by the cheaper coal-tar creosote (carbolic acid).



To obtain the creosote prepared according to this process permanently colorless, it is mixed with  $\frac{1}{4}$  to  $\frac{1}{2}$  per cent. of potassium dichromate and  $\frac{1}{2}$  to 1 per cent. of sulphuric acid, allowed to repose for 24 hours and again distilled. The small yield of creosote and its limited use make its profitable manufacture rather doubtful, except where sulphuric acid and soda can be procured at cheap rates.

The heavy oils are worked up in the same manner. The solution formed after treatment with soda solution is not added to the crude wood-vinegar, but treated by itself, as it contains scarcely any sodium acetate, but the sodium salts of the fatty acids with higher boiling points, such as propionic, butyric, valeric and caproic acids. This lye is used either for the preparation of these acids, or the solution is evaporated to dryness and ignited with the admittance of air to regain the soda.

If the acids are to be prepared, the solution is evaporated to the consistency of syrup, slightly oversaturated with sulphuric acid, and the resulting fluid diluted with water. The oleaginous layer collecting upon the surface consists of a mixture of the above-mentioned acids which are soluble with difficulty in water. By rectifying the mixture at the temperatures corresponding to the boiling points of the various acids, the latter are obtained in an almost pure state.

It is still more suitable to distil the mass previously evaporated to the consistency of syrup with alcohol and sulphuric acid, whereby the odoriferous ethers of the various acids are formed, which can then be separated by fractional distillation.

Since the heavy tar oils are entirely free from acid, and do not gum in the air, they may be used as lubricants for machinery, and were formerly much sought after for that purpose; but at present they have been largely superseded by petroleum products, and in consequence of this are of less value.

In the northern parts of Sweden and Finland considerable quantities of birch-tar oil are prepared, and below are given

the results of a series of experiments regarding the products which were obtained in the distillation of a sample of Finland birch-tar oil.

No. of the distillate.	Limits of boiling points.	Specific gravity.
1 . . . . .	212° to 226° F.	0.887
2 . . . . .	356° to 437° F.	1.020
3 . . . . .	588° to 644° F.	—

No. 1 formed a red-yellow, very mobile oil of a not disagreeable odor of birch tar. No. 2 was of a darker color and of a less agreeable odor, while No. 3 represented a dark brown, very viscous mass. By heating the residue in the still to above 644° F., it is suddenly decomposed, heavy vapors being evolved and a lustrous, very porous coal remaining behind.

By distilling the tar oil with caustic soda quite a series of oleaginous distillates are obtained.:

No. of the distillate.	Limits of boiling points.	Specific gravity.
1 . . . . .	212° to 284° F.	1.046
2 . . . . .	284° to 392° F.	1.114
3 . . . . .	392° to 437° F.	1.171
4 . . . . .	437° to 482° F.	1.058
5 . . . . .	482° to 734° F.	1.039

Nos. 1 to 3 were pale, red-yellow oils; Nos. 4 and 5 darker and more viscous. The residue remaining in the still at above 734° F. was a dark black mass, soft and flexible at the ordinary temperature, and becoming hard only at a lower temperature.

## PART II.

### MANUFACTURE OF CIDERS, FRUIT-WINES, ETC.

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#### CHAPTER XXVII.

##### INTRODUCTION.

THE term wine in general is applied to alcoholic fluids which are formed by the fermentation of fruit juices, and serve as beverages. According to this definition, there may actually be as many kinds of wine as there are fruits whose juices, in consequence of their content of sugar, are capable of vinous fermentation; and, in fact, besides the apple and pear, there are many other fruits which are likewise applicable to wine-making. Among these may be named, currants, gooseberries, mulberries, elderberries, cherries, oranges, dates, pine-apples, raspberries, strawberries, etc. But, in order to make the product from such fruits resemble the standard wine made from grapes, various ingredients have to be added, as, for instance, an acid, spices, coloring, and an astringent, to replace the extractive matter. Tartaric acid is, as a rule, used as an acid addition, and elderberry or whortleberry juice as coloring matter. The water employed in the manufacture should be pure and soft.

*Ripening of fruits.*—In order to form a clear idea of the process which takes place during the growth, ripening, and final decomposition of a fruit, it is necessary to refer to the constituents which are found in an unripe fruit at its first appearance.



Besides water, the quantity of which varies between 90 and 45 per cent., fruits contain partly soluble and partly insoluble substances. The juice obtained by pressure contains the soluble constituents, such as sugar, gum, tannin, acids, salts, etc., while the remaining insoluble portion consists chiefly of cellulose, starch, a gum-like body, a few inorganic substances, and further, the characteristic constituent of unripe fruits, to which the term *pectose* has been applied. It forms the initial point for the phenomena observed during the growth and ripening of fruits, and, therefore, requires a somewhat closer examination.

In regard to its behavior, pectose approaches cellulose and starch. It is chiefly found in the pulp of unripe fruits, but also in certain roots, especially in carrots, beets, and others. It is insoluble in water, spirits of wine, and ether, but during the ripening of the fruit it undergoes a change, induced by the acids and heat, and is converted into pectine, which is readily soluble in water. To pectose are due the hardness of unripe fruits and also the property of many fruits and roots of boiling hard in water containing lime, the pectose combining with the lime.

The formation of pectine commences as soon as the fruits are exposed to the action of heat, and then depends on the influence of the vegetable acid present upon the pectose. This can be shown by expressing the pulp of an unripe apple. The juice thus obtained contains scarcely a trace of pectine, but, by boiling it for a few minutes with the pulp of the fruit, the fluid, in consequence of the formation of pectine, acquires a viscous quality, like the juice obtained from ripe fruits.

Pectine, nearly pure, is white, soluble in water, non-crystallizable, and without effect upon vegetable colors. From its dilute solution it is by alcohol separated as a jelly, and from its more concentrated solution, in long threads. Brought into contact with alkalis or alkaline earths, pectine is transformed into pectic acid. Under the influence of a peculiar ferment called pectase, which will be described later on, pectine is

transformed into pectosic acid, and by dilute acids into metapectic acid.

By boiling a solution of pectine in water for a few hours, it partially loses its viscous condition and separates a substance called parapectine, which shows the same behavior as pectine, except that it is not precipitated by neutral lead acetate. When treated with dilute acids the parapectine is transformed into metapectine, which might be called metapectous acid, as it shows a decidedly acid reaction and colors litmus paper strongly red.

Metapectine is soluble in water, non-crystallizable, and, like pectine and parapectine, insoluble in alcohol, which precipitates it from its solutions in the form of a jelly. On being brought into contact with bases it is also transformed into pectic acid. It differs from pectine and parapectine in that the solution is precipitated by barium chloride.

Pectase, the peculiar ferment previously referred to, is similar in its mode of action to diastase and emulsion. It can be obtained by precipitating the juice of young carrots with alcohol, whereby the pectose, which was at first soluble in water, becomes insoluble, without, however, losing its effect upon the pectous substances.

By adding pectase to a solution of pectine, the latter is immediately converted into a jelly-like body, insoluble in water. This phenomenon is the *pectous fermentation*, which may be compared with lactic acid fermentation. It is not accompanied by an evolution of gas, and may take place with the air excluded, a temperature of 86° F. being most favorable for its progress.

Pectase is an amorphous substance. By allowing it to stand in contact with water for a few days, it decomposes, becomes covered with mold-formations, and loses its action as a ferment, this action being also destroyed by continued boiling. In the vegetable organism it occurs in a soluble as well as insoluble state.

Roots such as carrots, beets, etc., contain soluble pectase,

and their juice added to a fluid containing pectine in solution immediately induces pectous fermentation, while the juice of apples and other acid fruits produces no effect upon pectine, the latter being present in them in a modified insoluble form and accompanying the insoluble portion of the pulp. On adding the pulp of unripe apples to a pectine solution it gelatinizes in a short time, in consequence of the formation of pectosic and pectic acids. It is therefore due to the presence of these acids that many ripe fruits are so easily converted into jellies.

Pectosic acid is the result of the first effect of the pectase upon pectine; it being, however, also formed by bringing dilute solutions of potash, soda, ammonia or alkaline carbonates in contact with pectine. In all these cases salts are formed which, when treated with acids, yield pectosic acid. The latter is gelatinous, and with difficulty dissolves in water. In the presence of acids it is entirely insoluble. It is quickly transformed into pectic acid by long boiling in water, by pectase, or by an excess of alcohol.

By allowing pectase to act for some time upon pectine, pectic acid is formed; the same conversion taking place almost instantaneously by dilute solution of potash, soda, ammonia, alkaline carbonates, as well as by barium, lime and strontium water. Its formation in the above-described manner is preceded by that of pectosic acid, which, as previously mentioned, is converted by the same agents into pectic acid.

Pectic acid is insoluble in cold, and scarcely soluble in hot, water. By boiling it, however, for a certain time in water, and constantly replacing the water lost by evaporation, it disappears entirely, and is converted into a new acid, soluble in water. Alkalies decompose it very rapidly, the final result being metaplectic acid, which is soluble in water, but non-crystallizable. On boiling in hot water, the solution forms, after cooling, a jelly.

Pectic acid further possesses the special property of dissolving in a large number of alkaline salts and forming with them



true double salts, which always show a decidedly acid reaction, dissolve in water, and on cooling form consistent jellies.

By boiling for a few hours a solution of a pectous salt, the latter is transformed into a parapectous salt which, when decomposed by a dilute acid, yields parapectic acid. It is non-crystallizable, shows a strong acid reaction, and forms with alkalis soluble salts. It is precipitated by barium water in excess.

Metapectic acid is formed in various ways, among others by leaving an aqueous solution of parapectic acid to itself for some time, but also by the action of the lime contained in the cell-tissues of roots and fruits upon pectose. It is insoluble in water, does not crystallize, and gives soluble salts with all bases. With an excess of bases the salts acquire a yellow coloration. They are precipitated by basic lead acetate.

What has been said in the preceding may be briefly condensed as follows:—

1. By the influence of heat upon pectose pectine is formed.
2. Pectine is transformed into parapectine by boiling its aqueous solution for several hours.
3. Parapectine, when treated at a boiling heat with dilute acids, is converted into metapectine.
4. Pectase converts pectine into pectic acid.
5. By long-continued action of pectase upon pectine, pectic acid is formed.
6. Pectic acid is by boiling water transformed into parapectic acid.
7. An aqueous solution of parapectic acid is rapidly converted into metapectic acid.

All these bodies are derived from pectose, which through all these transformations has not even suffered a change in the proportion of weight of its constituents (carbon, hydrogen, and oxygen); and hence all have the same qualitative and quantitative compositions. This may, perhaps, sound odd, but chemistry presents numerous analogies for such cases, and hence the term *isomeric* has been applied to bodies which, with

the same quantitative composition, exhibit very different chemical properties.

The changes pectose undergoes by the influence of heat, by the action a peculiar ferment, acid and alkalies, and the resulting combinations mentioned above, have of course been artificially effected by chemical means. They resemble, however, so closely the state of fruits in the course of their growth and ripening, and the influences and conditions to which fruits are exposed in nature are sufficiently similar to those artificially induced, that their action may be reasonably supposed to be the same. We know from daily experience that heat promotes the development and ripening of fruit. Fruits contain pectose and acids, and alkalies and bases are conducted to them from the soil; and hence in fruit in a normal state of development none of the chemical agents are wanting which the chemist uses for the production of derivatives of pectose.

If the transformation of substances under the influence of other substances be considered as dependent on chemical processes, the development of a fruit from its first formation to complete ripeness, and even to its decomposition, rotting, and putrefaction, is a chemical process in the widest sense of the word. This is evident, not only from what has been said in the preceding, but has also been plainly shown by special chemical researches into the changes fruits undergo during their development and perfection. The results of these researches are briefly as follows:—

1. The quantity of water contained in the pulp of a fruit is considerable; it varying between 45 and 90 per cent. In many fruits the content of water remains unchanged during the different periods of ripening, but, as a rule, it is somewhat greater in the commencement.

2. Fruits of the same kind examined at the same season of the year always contain the same quantity of water; the same holding good as regards the various parts of the pulp of a fruit.

3. The solid constituents in the pulp of fruits amount to between 10 and 25 per cent. They consist of soluble substances

which dissolved in the water from the juice of the fruits ; and of insoluble bodies which compose the membranes of the cells.

4. The quantity of soluble substances always increases with increasing ripeness, while the weight of the insoluble decreases ; and hence it may be said that the soluble substances contained in the juice of a fruit are formed at the expense of the insoluble portion of the pulp. The bodies which become soluble are starch, pectose, and a gum-like substance capable of being converted into gum.

On this modification of the solid portion of the pulp of a fruit depend also the changes a fruit undergoes in regard to hardness and transparency during ripening.

According to the mode of action of the pectase and acids upon the pectose, all ripe fruits contain pectine.

5. Various acid fruits, such as plums, cherries, etc., are frequently observed to secrete a neutral juice which, in consequence of the evaporation of the water, leaves a gum-like substance upon the exterior of the fruit. This phenomenon throws some light upon the separation of gum as it appears in many trees, and which, when it occurs very abundantly, is actually a disease.

In fruits becoming thus covered with a gum, a transparent, neutral substance insoluble in water occurs stored in the cells of the pulp. Under the influence of nitrogenous substances, which act as a ferment, and perhaps also of acids, this gum-like substance is modified and transformed into actual gum, which is then converted into sugar in the interior of the pulp of the fruit ; an excess of this gum-like substance being secreted and forming a firm coating upon the exterior of the fruit.

6. The sugar occurring in ripe fruits is evidently derived from various sources. The occurrence of a large quantity of starch in many unripe fruits, especially in apples, and its complete disappearance at the time of ripeness, allow of no other explanation than that the sugar occurring in fruits is formed by the conversion of the starch under the influence of the acids present ; other indifferent substances, such as gum, vegetable



mucus, etc., undergoing similar transformations and yielding in this manner a certain portion of sugar. Even tannin, which occurs in all unripe, but not in ripe, fruits, can be changed by acids and ferments so as to form sugar.

Thus far nothing justifies the supposition that the acids in fruits, such as tartaric, citric, malic acids, are converted into fruit sugar. To entertain such an opinion it would have to be supposed that the molecules of these acids, which are far more simple than those of fruit-sugar, become more complex and are converted into sugar. In such natural transformations the reverse is, however, generally the case, the molecules always endeavoring to become the more simple the farther they withdraw from organized structures.

7. It has been attempted to explain in various ways the very remarkable phenomenon of the gradual disappearance of the acid in ripening fruits. It might not be impossible that the acid of a fruit is neutralized by the bases conducted to it through the juice; or that it is covered by the sugar or the mucous substances formed in the juice; or, finally, that it disappears at the moment of ripeness by suffering actual combustion. An examination of these various theories leads to the conclusion that the acid is neither neutralized nor covered by the sugar or the mucous substances, but that it actually undergoes slow combustion.

During the stages of development and ripening, a fruit passes through two different stages sharply separated from each other by definite chemical phenomena. In the first stage, which may be designated as that of growth, whilst the fruit remains green, its relation to the atmosphere appears the same as that of leaves, for it absorbs carbonic acid and evolves oxygen. During this epoch it increases rapidly in size, and receives through the stem the inorganic substances, indispensable for its development, and the water. If, at this stage, it is taken from the tree, it soon commences to wither and decay. But in the second period, when it fairly begins to ripen, its green color is, as a rule, replaced by a yellow, brown-red, or

red. Oxygen is now absorbed from the air and carbonic acid is evolved, whilst the starch and cellulose are converted into sugar under the influence of the vegetable acids, and the fruit becomes sweet. When the sugar has reached the maximum the ripening is completed, and if the fruit be kept longer, the oxidation takes the form of ordinary decay.

## CHAPTER XXVIII.

### FRUITS AND THEIR COMPOSITION.

For the preparation of fruit-wines, not only the fruits cultivated in our gardens and orchards on account of their fine flavor are used, but sometimes also others which do not by any means possess an agreeable taste, and whose juices, after fermentation, yield a product which has at least only a very doubtful claim to the name of "wine." The utilization of such material for wine-making can only be explained by special fancy, and hence here only such fruits will be considered as, on account of the nature of their juices, will yield with rational treatment a beverage of a sufficiently agreeable taste to be liked.

For making fruit-wine, sugar not only by itself but also in its proportion to the free acid present, is undoubtedly the most important constituent of the fruit. The following table from Fresenius gives the average percentage of sugar in different varieties of fruit:

#### I.

Peaches . . . . .	1.57 p. c.	Currants . . . . .	6.10 p. c.
Apricots. . . . .	1.80 "	German prunes . . . . .	6.25 "
Plums. . . . .	2.12 "	Gooseberries . . . . .	7.15 "
Reine Claudes . . . . .	3.12 "	Pears. . . . .	7.45 "
Greengages . . . . .	3.58 "	Apples. . . . .	8.37 "
Raspberries . . . . .	4.00 "	Sour cherries . . . . .	8.77 "
Blackberries . . . . .	4.44 "	Mulberries . . . . .	9.19 "
Strawberries . . . . .	5.73 "	Sweet cherries. . . . .	10.79 "
Whortleberries. . . . .	5.78 "	Grapes. . . . .	14.93 "

II. Table according to average percentage of free acid expressed in malic acid :

Pears . . . . .	0.07 p. c.	Blackberries . . . . .	1.19 p. c.
Greengages . . . . .	0.58 "	Sour cherries. . . . .	1.28 "
Sweet cherries . . . . .	0.62 "	Plums. . . . .	1.30 "
Peaches . . . . .	0.67 "	Whortleberries. . . . .	1.34 "
Grapes . . . . .	0.74 "	Strawberries . . . . .	1.37 "
Apples . . . . .	0.75 "	Gooseberries . . . . .	1.45 "
German prunes. . . . .	0.89 "	Raspberries . . . . .	1.48 "
Reine Claudes . . . . .	0.91 "	Mulberries. . . . .	1.86 "
Apricots. . . . .	1.09 "	Currants. . . . .	2.04 "

III. Table according to the proportion between acid, sugar, pectine, gum, etc.

	Acid.	Sugar.	Pectine, gum, etc.
Plums . . . . .	1	1.63	3.14
Apricots . . . . .	1	1.65	6.35
Peaches . . . . .	1	2.34	11.94
Raspberries . . . . .	1	2.70	0.96
Currants. . . . .	1	3.00	0.07
Reine Claudes . . . . .	1	3.43	11.83
Blackberries . . . . .	1	3.73	1.21
Whortleberries . . . . .	1	4.31	0.41
Strawberries . . . . .	1	4.37	0.08
Gooseberries . . . . .	1	4.93	0.76
Mulberries . . . . .	1	4.94	1.10
Greengages . . . . .	1	6.20	9.92
Sour cherries . . . . .	1	6.85	1.43
German prunes . . . . .	1	7.03	4.35
Sweet cherries . . . . .	1	11.16	5.60
Grapes . . . . .	1	20.18	2.03
Pears . . . . .	1	94.60	44.40

IV. Table according to the proportion between water, soluble and insoluble substances.



Composition of the juice in  
100 parts, without the in-  
soluble substances.

	Water.	Soluble substances.	Insoluble substances.	Water.	Soluble substances.
Raspberries . . . . .	100	9.12	6.88	91.64	8.36
Blackberries . . . . .	100	9.26	6.46	91.53	8.47
Strawberries . . . . .	100	3.39	5.15	91.42	8.58
Plums . . . . .	100	9.94	0.87	91.13	8.87
Currants . . . . .	100	11.00	6.62	90.09	9.91
Whortleberries. . . . .	100	12.05	16.91	89.25	10.75
Gooseberries . . . . .	100	12.18	3.57	89.14	10.86
Greengages . . . . .	100	13.04	1.53	88.46	11.54
Apricots. . . . .	100	13.31	2.07	88.25	11.75
Pears . . . . .	100	14.25	5.54	87.52	12.48
Peaches . . . . .	100	14.64	2.10	87.23	12.77
German prunes. . . . .	100	15.32	3.15	86.71	13.29
Sour cherries . . . . .	100	16.48	1.31	85.85	14.15
Mulberries . . . . .	100	16.57	1.47	85.79	14.21
Apples . . . . .	100	16.89	3.61	85.46	14.54
Reine Claudes . . . . .	100	18.52	1.22	84.37	15.63
Sweet cherries . . . . .	100	18.61	1.53	84.30	15.70
Grapes . . . . .	100	22.81	5.81	81.42	18.58

V. Composition of the juice according to the content of  
sugar, pectine, etc., in 100 parts :

	Sugar, p. c.	Pectine, etc., p. c.		Sugar, p. c.	Pectine, etc., p. c.
Peaches . . . . .	1.99	10.05	German prunes . . . . .	7.56	4.70
Reine Claudes . . . . .	2.04	6.98	Gooseberries . . . . .	8.00	1.24
Apricots. . . . .	2.13	8.19	Whortleberries . . . . .	8.12	0.77
Plums. . . . .	2.80	5.40	Pears. . . . .	8.43	4.02
Greengages. . . . .	4.18	6.45	Apples . . . . .	9.14	4.59
Raspberries . . . . .	4.84	1.73	Mulberries . . . . .	10.00	2.22
Blackberries . . . . .	5.32	1.72	Sour cherries . . . . .	10.44	2.17
Strawberries . . . . .	6.89	0.13	Sweet cherries. . . . .	15.30	2.43
Currants. . . . .	7.30	0.16	Grapes. . . . .	16.15	2.07

VI. Content of free acid in 100 parts of juice :

Pears . . . . .	0.09	p. c.	Blackberries . . . . .	1.42	p. c.
Reine Claudes . . . . .	0.59	"	Sour cherries. . . . .	1.52	"
Greengages . . . . .	0.67	"	Strawberries . . . . .	1.57	"
Grapes . . . . .	0.80	"	Gooseberries . . . . .	1.63	"
Apples . . . . .	0.82	"	Plums. . . . .	1.72	"
Peaches . . . . .	0.85	"	Raspberries . . . . .	1.80	"
Sweet cherries . . . . .	0.88	"	Whortleberries. . . . .	1.88	"
German prunes. . . . .	1.08	"	Mulberries. . . . .	2.02	"
Apricots. . . . .	1.29	"	Currants. . . . .	2.43	"

Tables V. and VI. represent the proportion in which the soluble constituents of the fruit are found in the juice or must obtained from them. In the practical execution of the preparation of fruit wines we will have occasion to refer to these tables.

For the preparation of wine, only the soluble substances, which pass into the must, and from which the wine is formed, are chiefly of interest, and it will be necessary to consider them somewhat more closely.

*Grape-sugar or Glucose.*—This sugar is widely diffused throughout the vegetable kingdom, it occurring in most kinds of sweet fruits, in honey, etc. Artificially it can be readily obtained by heating a solution of cane-sugar with a dilute acid. It is also formed by dissolving cane-sugar in wine. On a large scale it is prepared by boiling starch with very dilute sulphuric acid for several hours, neutralizing the liquid with chalk and evaporating the solution.

Grape-sugar is much less sweet than cane-sugar. In alcohol of 90 per Tr. it is sparingly soluble; in hot water it dissolves in every proportion; of cold water it requires, however, about  $1\frac{1}{3}$  parts for solution. It crystallizes from aqueous solution with one molecule of water, in cauliflower-like masses and from hot alcohol in warty, anhydrous needles. A solution of crystallized grape sugar turns the plane of polarization to the right, but one of anhydrous grape-sugar to the left.

*Acids.*—The acid reaction of fruit juices is partly due to malic acid and partly to citric acid, and also as in the case of grapes to tartaric acid. As a rule all these acids are present; in currants citric acid predominates; in apples, etc., malic acid.

The presence of potassium in grape-must gives rise to the formation of potassium bitartrate of crude tartar. Tartar requires for its solution 240 parts of cold water; in alcoholic fluids it is less soluble, and hence it is found as a crystalline deposit in wine casks. Fruit-musts contain no tartaric acid, and, consequently, the wines prepared from them cannot de-

posit tartar. The salts formed by malic and citric acids with potassium being readily soluble and even deliquescent form no deposit in the wine.

*Albuminous substances.*—By this general term are designated several nitrogenous vegetable substances which have the same composition ; they being vegetable albumen, fibrin, and glue. The quantities of these substances in the different musts are, on the one hand, so small, and the difficulty of accurately distinguishing them from each other is, on the other, so great, that it is scarcely possible to definitely determine the kind actually present in the fruit juice. Most likely all are present at the same time.

For the preparation of wine these bodies are of importance ; they furnishing the material for the development of the yeast-fungus during fermentation.

*Pectous substances.*—Under the heading “ Ripening of fruits,” the pectous substances have been sufficiently discussed. They are scarcely ever wanting in a fruit juice, but being insoluble in alcoholic fluids they are entirely separated with the yeast, and hence are not present in fruit-wines.

*Gum and Vegetable Mucilage.*—Our knowledge as regards gum is still limited. Gum-arabic, which may be studied as a representative of this class, is an exudation from certain species of acacia and consists essentially of arabin. It is generally supposed to be soluble in water, but on endeavoring to filter a somewhat concentrated solution not a drop will be found to run off, and the little which possibly may pass through the filter is by no means clear.

Closely related to gum-arabic is bassorine, the gum which exudes from the cherry, plum, almond, and apricot trees. It does not give a slime with water, but merely swells up to a gelatinous mass.

Wine brought in contact with the smallest quantity of gum-arabic remains permanently turbid and cannot be clarified by filtering or long standing. From this behavior of gum it may be concluded that, though it may occur dissolved in the must, it is not present in the wine.



The various kinds of vegetable mucilage have also not yet been accurately examined ; it only being known that there are quite a number of them. It is, however, likely that only a few of them are actually soluble in water. Though the mucilage of certain seeds, such as linseed and quince-seed, may be considered to be as soluble in water as gum-arabic, and perhaps more so, because it is a perfectly clear fluid drawing threads, yet on filtering it will be found that what passes through contains scarcely a trace of mucilaginous substance. Hence, it is doubtful whether mucilages exist which are actually soluble in water, and whether they occur in wine. Artificial dextrin is, however, an exception, as it forms with water a perfectly clear fluid, which can be filtered. Attention may here be called to an easy method of distinguishing between solution of gum-arabic and of dextrin. The first cannot be heated, even for a minute, over an open fire without scorching, while the latter can be completely boiled down without fear of burning.

*Tannin*.—Several kinds of tannin occur in plants, which can, however, be finally reduced to two modifications, viz.: *pathological* and *physiological tannin*. The first occurs in large quantity in nut-galls, especially in the Chinese variety, also in sumach (the twigs of *Rhus coriaria*) and in many other plants. Pathological tannin is characterized by splitting under the influence of dilute acids as well as by fermentation into gallic acid and grape-sugar. Furthermore, it completely precipitates glue from its solutions, but it is not suitable for the conversion of the animal skin into technically serviceable leather which will withstand putrefaction. Besides, only the gallic acid obtained from pathological tannin yields pyrogallic acid by destructive distillation.

Physiological tannin is chiefly found in materials used for tanning. It cannot be split by dilute acids or fermentation, does not yield gallic acid, and the product of destructive distillation is not pyrogallic acid, but pyrocatechin or oxyphenic acid. It converts the animal skin into perfect leather.

There can be but little doubt that physiological tannin is the variety found in fruits and fruit-juices. Generally speaking, a content of tannin in wine is not exactly a desirable feature, as it is readily decomposed. It can only have an advantageous effect when the wine contains an excess of albuminous substances which the tannin removes by entering into insoluble combinations with them. This may be the reason why wine containing tannin is considered more durable, because if it contained albuminous substances in large quantity it would be still more readily subjected to changes. Under such circumstances a small addition of tannin to the wine may be of advantage, though instead of tannin it is advisable to use an alcoholic extract of grape-stones, they being uncommonly rich in tannin.

*Inorganic constituents.*—The inorganic constituents of the different varieties of fruit are very likely the same, namely, potash, lime, magnesia, and sulphuric and phosphoric acids, they varying only in the proportions towards one another and in the total quantity of all the substances. Moreover, their quantity is too small to exert an influence upon the quality of the wine to be produced, being of interest only in regard to the exhaustion of the soil. Though lime and sulphuric acid in sufficient quantity occur almost everywhere in the soil, this cannot be said of potash and phosphoric acid. Unfortunately there are no accurate statements regarding the amount of these substances which is withdrawn from the soil by the crop of one year, but there can be no doubt that it is very large, and that consequently fruit trees from time to time require a certain amount of manure in order to return to the soil what has been taken from it.

*Fermentation.*—Fermentation is a chemical process which is always caused by the presence of a ferment or a substance in a peculiar state of decomposition. Although to induce fermentation the presence of a ferment is necessary, it does not take part in the decomposition of the fermenting substance. The products of fermentation vary according to the nature of the

fermenting body, as well as according to the nature of the ferment itself. Each peculiar kind of fermentation requires a certain temperature, and it is nearly always accompanied by the development of certain living bodies (bacteria or fungi).

When yeast is added to a dilute solution of dextrose or another glucose, vinous fermentation speedily sets in; whilst a solution of cane-sugar undergoes fermentation but slowly, the cause being that this sugar must first be converted into inverted sugar before fermentation can commence. Vinous fermentation proceeds most rapidly at 77° to 86° F., and does not take place below 32° or above 95° F. The presence of a large quantity of acids or alkalies prevents fermentation, while if the liquid has a faint acid reaction, fermentation proceeds best.

The yeast which is formed in the fermentation of the juice of grape and other kinds of fruit is produced from soluble albuminous bodies contained in fruit. It consists of one of the lowest members of the vegetable kingdom (*Torula cerevisiæ*), and under the microscope is seen to be made up of little oval transparent globules, having a diameter of not more than 0.1 millimeter and often adhering in clusters and strings. They are propagated by budding, and die as soon as they have reached their highest state of development. In contact with air and water yeast soon undergoes putrefaction.

The chief products of vinous fermentation are alcohol and carbon dioxide; a small quantity of the sugar being at the same time converted into other products, about 2.5 per cent. being transformed into glycerin and 0.6 to 0.7 per cent. into succinic acid. A further portion of the sugar, about one per cent., is assimilated in the form of cellulose by the yeast and separated. By the simultaneous formation of these different secondary products about 5.5 to 6.5 per cent. of sugar is lost in the formation of alcohol. As they are not always formed in equally large quantities, no conclusion can be arrived at from the content of sugar in the must as to the quantity of alcohol corresponding to theory in the finished wine. It is, as a rule, supposed that the sugar yields one-half its weight



of alcohol, which is sufficiently correct for all practical purposes.

Absolute alcohol, *i. e.*, alcohol entirely free from water, is a very mobile fluid, clear as water and almost odorless. It boils at  $173^{\circ}$  F., and when it is cooled down to  $148^{\circ}$  F. it becomes viscid, but does not solidify. Its specific gravity at  $32^{\circ}$  F. is 0.80625, and at  $59^{\circ}$  F. 0.79367. It is very inflammable, and burns with a blue, non-luminous flame. It absorbs moisture with great avidity, and is miscible with water in all proportions, the mixture evolving heat and undergoing contraction.

The methods for determining the content of alcohol in a fluid have already been previously given.

*Succinic acid*.—No accurate researches have as yet been made in regard to the quantity of this acid in wine, its influence upon the quality of the wine, and the conditions under which more or less of it is formed during fermentation. According to Pasteur, the more succinic acid is formed the slower fermentation progresses; the weaker the development of yeast and the less nourishment offered to the latter. In acid fluid more succinic acid is formed than in neutral.

Succinic acid is quite readily soluble in a mixture of alcohol and water, and consequently also in wine. Its taste is not very sour, but disagreeable, and adheres for some time to the tongue; hence its presence can scarcely be expected to give an agreeable taste to the wine.

*Glycerin*.—Glycerin being found in grape-wines, in which it is formed from the sugar by fermentation, there can scarcely be any doubt of its formation under the same conditions in fruit wines. According to Pasteur, the quantity of glycerin in wine is in a definite proportion to the succinic acid formed, and, hence, more glycerin would be produced with slow fermentation and in an acid fluid. In red wines Pasteur found 4 to 7 per cent. of glycerin.

Pure glycerin is a colorless, very viscid liquid having a specific gravity of 1.27. It can be mixed with water and alcohol in all proportions and possesses a very sweet taste. It is

very likely that the mild sweet taste of many ripe wines is due to a certain content of glycerin.

A solution of 7 parts of glycerin in 1000 of water (the proportion in which Pasteur found glycerin in wine) does not possess a sweet taste and differs from water only in being more insipid. By adding to such a solution 100 parts of alcohol, the mixture shows a taste different from that of alcohol alone, diluted in the same proportion, the predominant taste of the latter being decreased by the glycerin and that of the mixture becoming milder. Hence a certain importance has to be ascribed to the glycerin.

*Carbonic acid.*—The greater portion of the carbonic acid formed by fermentation escapes as a gaseous body during the process, but a certain portion remains dissolved in the wine as long as the temperature of the latter is not raised. The temperature of cellars generally increases, however, towards the end of spring, which causes anew a slight development of carbonic acid in consequence of which the wine again becomes turbid. The presence of carbonic acid is of advantage only in young wine, as it protects it from the direct action of the air by forming a layer upon the surface. In old wines it conceals, however, the fine aroma and taste, making them appear younger than they actually are.

Though it cannot be said that carbonic acid plays an essential part in the preparation of wine, it deserves attention on account of its deleterious influence upon the workmen. To avoid all injurious consequences, provision should be made for a thorough ventilation of the cellar by means of windows and doors. If fermentation is carried on in barrels, the carbonic acid developed in a number of them should be conducted by means of tubes secured air-tight in the bungs to a zinc pipe which passes through a suitable aperture into the open air.

*Alkaloid in wine.*—It has frequently been asserted that an alkaloid exists in young wine, which not being contained in the must or the yeast must have been formed from the nitrogenous constituents of the yeast or of the fluid during fermen-

tation. It has not been found in old wine, and it is therefore concluded that it in time decomposes. Should this observation be confirmed, it would explain the difference in the effects of the very intoxicating young wines and of old wines.

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## CHAPTER XXIX.

### MANUFACTURE OF CIDER.

THE first step in the preparation of fruit-wines is to obtain the juice or must from the fruit. Stamping or grinding and subsequent expressing of the paste thus formed by means of strong pressure suffice in most cases for berries and other small fruits. With apples, etc., this manner of reduction is not only difficult, but also connected with considerable loss caused by larger and smaller pieces jumping from the trough.

The earliest appliance known was simply a trough in which the apples were reduced to an imperfect pomace by rolling them with a heavy cylindrical stone or by pounding them as in a mortar. An improvement was the production of the English cider-mill. This consisted of a pair of coarsely corrugated iron cylinders from which the apples fell to a second pair close together and finer in their surfaces, and passed through finely mashed to the pomace vessel underneath. In 1852, Mr. W. O. Hickock, of Harrisburg, Pa., invented a portable cider-mill which consisted of a pair of small horizontal cylinders armed with small spirally arranged teeth or spikes revolving close together, one at a higher velocity than the other. The apples were first broken by the action of a coarsely-fluted roller which revolved against a table under the hopper, and after passing between the cylinders, the apples were not only bruised but also grated into the required pomace. This machine was capable of grinding 100 bushels of apples per day. Numerous modifications have been made in the



plan of Mr. Hickock's mill, some being simply spiked cylinders against which the apples were carried and held till grated by reciprocating plungers.

The limits of this work will not permit of a notice of all the various styles of portable mills before the public or the multitude of graters or apple grinders, many of which possess excellent points and are worthy of commendation. An excellent apparatus for crushing apples is the crushing-mill shown in Figs. 90 and 91, *B C* (Fig. 91) representing the cylinders provided with teeth. A hopper, *A*, receives the apples, which pass between the cylinders, where they are crushed and fall

FIG 90.

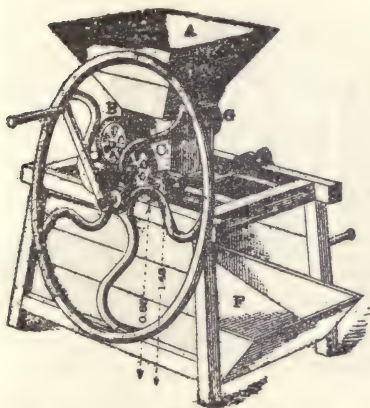
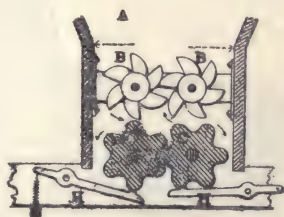


FIG. 91.

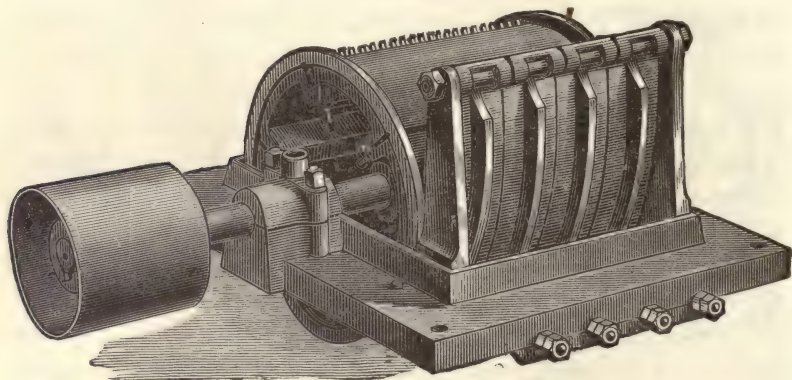


into the receiver *F* placed underneath. Two men operate this mill by means of cranks. Larger and stronger mills are used when the quantity of apples seems to require them, and in that case horse-power is applied.

Fig. 92 shows Davis's star apple-grinder. The grinder shown in the illustration is a heavy machine weighing 340 lbs. The cylinder is 12 inches in diameter and 12 inches long, is turned and carefully balanced, has grooves planed in to receive the knives, six in number, which are finely made and tempered. Each knife furnished is made of steel-plated iron, the steel being very thin and having a back of iron; there

is no danger of breaking, although made very hard. The end of the cylinder is banded with wrought-iron bands and the knives are set with set-screws. The shaft is of steel and runs in anti-friction metal. The concaves are hung at top, so they can swing back at the bottom to allow stone, pieces of iron, etc. to pass through without injuring the knives. The concaves are held to their places by a bolt which allows the concave to be set as close as desired to the cylinder, and is held to its place by coil-springs which will give enough to allow stones to pass and yet hold rigid in grinding even frozen apples. The frame is one casting, and as the concaves

Fig. 92.



are fast to the frame they cannot get out of line or be displaced, as in the case when the concave is fast to the hopper. The hopper can be readily removed to adjust knives, and all parts are adjustable and easy to get at. This machine can be gauged to grind from 200 to 400 bushels per hour. Power required to grind six bushels per minute, about six horse-power, say about as many horse-power as desired to grind bushels per minute.

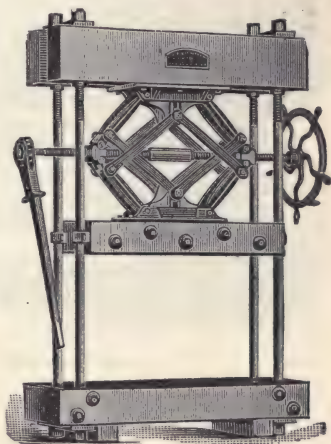
*Presses.*—For obtaining the juice from berries, etc., a press is generally not required, or at least only a slight pressure; the greater portion of it running out from the must by placing

the latter upon a cloth spread over a perforated bottom in a vat. The juice retained by the lees, which, as a rule, is very sour and has to be diluted with water, can be extracted with the latter more completely than is possible with the strongest press.

For obtaining the juice from apple pomace, etc. a good press is, however, an important auxiliary. Before the introduction of screws the method of extracting the juice of the apple was by the use of heavy weights, wedges, and leverage. Until within a late period a large wooden screw was used and is even now employed in some sections of the country. Of these screws two and frequently three and four, set in a strong framework of double timbers, were found no more than sufficient to separate the cider from the pomace. In order to operate these screws a long heavy wooden lever became necessary, which required the united services of four or five men to handle, and not unfrequently the strength of a yoke of oxen was called into requisition before the work could be accomplished. An improvement upon the wooden screw was made by the substitution of the iron screw and iron nut. But the objectionable feature of having to handle heavy and cumbersome levers still remained, making labor irksome and expensive. In modern presses this difficulty has been entirely overcome, and the juice is extracted from the pomace with great ease and completeness.

Of the many presses before the public, a hand-press and a power-press are here illustrated; presses of all sizes between these two are found in the market. Fig. 93 shows the "Farmer's cider-press." It is 7 feet 1 inch high, with a width between

Fig. 93.

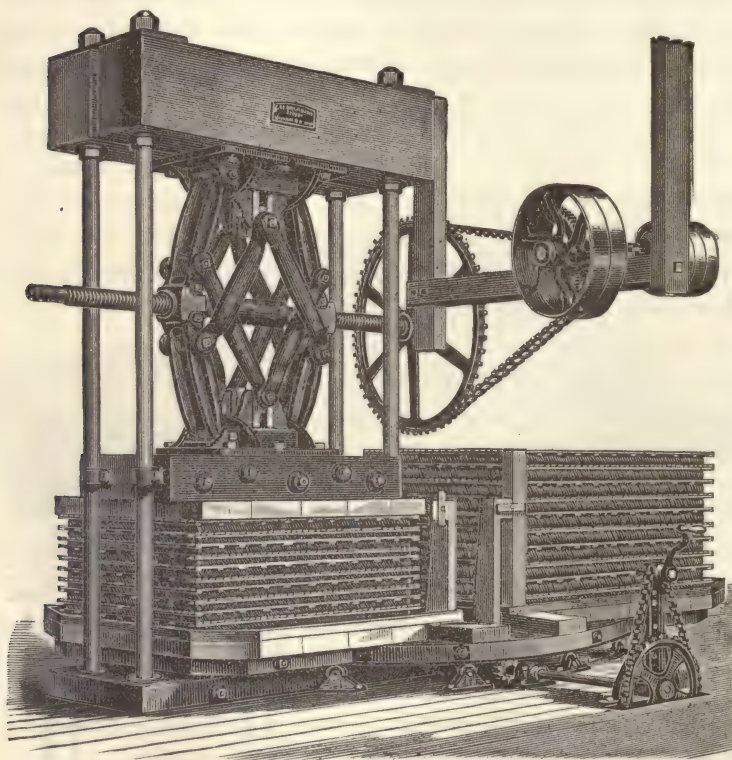




the rods of 3 feet  $1\frac{3}{4}$  inches. It will hold 15 to 16 bushels of apples at a pressing and is especially designed for individual use. It is also admirably adapted for squeezing the juice from small fruits, berries, etc.

Fig. 94 shows the "Extra power cider-press," with revolving platform. It is 13 feet 4 inches high, 6 feet 4 inches wide

FIG. 94.

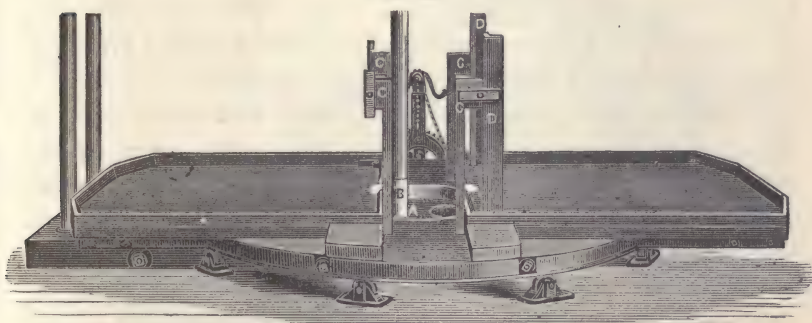


between the rods, and has a platform 13 feet 3 inches long. It gives a pressure of 250 tons. The press is always loaded in one place, and consequently the grater can be located immediately over the middle of the cheese, avoiding the necessity of conveying the pomace from one end of the press

to the other. This press can easily make a pressing of 12 barrels of cider each hour.

Fig. 95 shows the revolving platform belonging to the above press, for which the following advantages are claimed: 1. Both ends of the platform are loaded and unloaded in the same place. 2. It is so geared that one man can easily and quickly revolve it. 3. The grinder can be directly over the centre of the cheese, thus avoiding all the labor of shoveling the pomace. 4. The pomace being dropped in the centre of the cheese, it is an easy matter to spread it with equal density over the entire surface, thus building a cheese that is not liable to tilt or slide. The cider runs into a copper basin in

FIG. 95.



the centre of the platform between the two cheeses. The basin is so arranged that it receives the cider while the platform is being revolved as well as while the press is working.

*A* is the copper basin to receive the cider from platforms, and has an outlet through the bottom, about 6 inches in diameter, for the cider to pass off into the tank below. *B* is a copper tube encasing the rods. *C, C, C, C* are four posts fastened to the platform to hold guide-pieces for racks. *D, D* are rack guides.

*Improved Racks.*—The single racks are made of some light and tough wood—bass-wood or spruce seems best—cut into strips about  $\frac{1}{2} \times \frac{7}{8}$  inch and placed about  $\frac{1}{4}$  inch apart, with

four, five, or more elm strips, 2 inches wide about  $\frac{3}{8}$  inch thick, placed across and nailed to the narrow slats. The 2-inch slats extend beyond the narrow ones on each side about 4 inches. This is to support the wings, which are fastened to the rack by 3 or more bronze hinges. These wings, with the aid of 2 retaining bars, make the box to form the pomace in. The slats are rounded on the edges, so as not to injure the press-cloth. Steel wire nails or wire staples are used of sufficient length to clinch.

Double racks are made by using slats  $\frac{5}{16} \times \frac{7}{8}$  inch. The slats on one side are laid directly across the slats on the other side. Four wide slats are put at the outer edges, then these are all fastened together by steel wire nails or staples. These racks have the advantage of having an even surface on each side. The press-cloth will last much longer than when used on single racks, where it is strained over 4 to 9 elm slats.

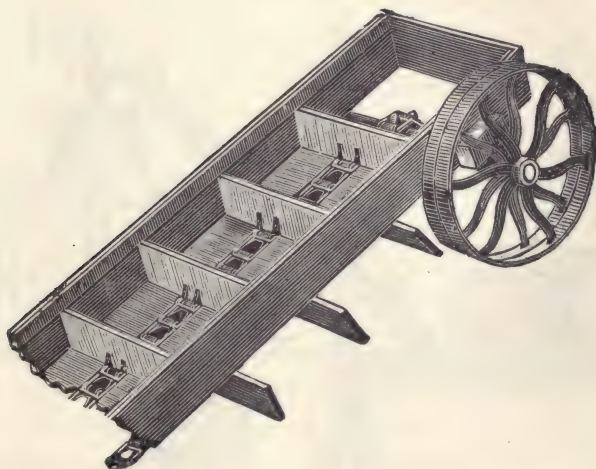
To lay up a cheese with the improved rack, commence on the platform of the press and lay a rack; then turn up the wings on each side of the rack and place the retaining bars on each end, with the hooks on the outside of the wings, so as to hold them up. Over this box spread the cloth, fill the box evenly full of pomace, then turn in the sides and ends of the cloth over the pomace, the cloth being of sufficient size to cover it. The retaining bars are then removed, allowing the wings to fall in place. Another rack is placed on the cheese just made, the retaining bars placed in position to hold up the wings, another cloth placed on the box, etc., and this operation is continued until there is the right number of layers in the press. A rack should be placed on the top of the last layer. A guide should be used in laying up the cheese, so as to bring each rack directly above the other.

*Plain racks.*—These are made, either single or double, of slats of the same description and dimensions as are used in the improved racks, but in the place of wings and retaining bars, a form square in size and 4 inches deep is used to form the sides of a box for the pomace. In laying up a cheese com-



mence by placing a rack on the platform, and upon this place the form, spread a cloth over the form and fill even up with pomace; then fold the ends and sides of the cloth over onto the pomace, as described with the other style of rack, and remove the form. Place another rack on the layer just formed, and put the form on that and proceed as before until the cheese is complete. It will require one cloth less than the number of racks used for a cheese. Care must be exercised in laying a cheese to have the racks come evenly, as they are liable to tilt if they overhang. The best way to avoid the

FIG. 96.



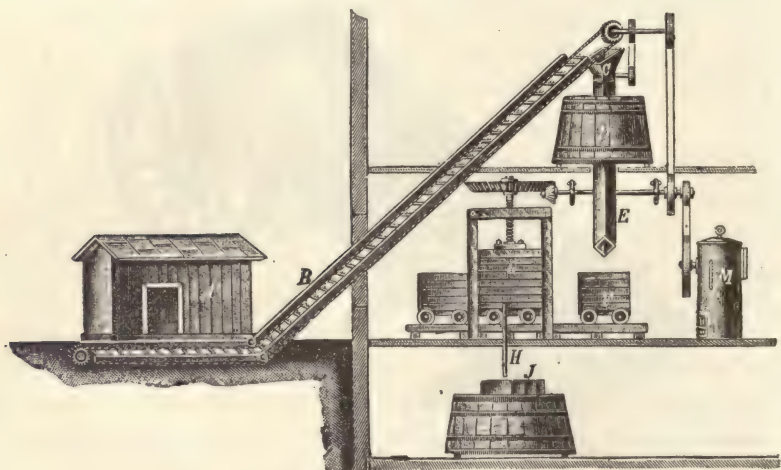
liability to slide or tilt is to lay the racks alternately the length and breadth of the press.

In the equipment of a first-class modern cider mill nothing gives better satisfaction for the money expended than an apple elevator. The expense is a small matter compared with the convenience of having the mill so arranged that apples may be brought from any part by a perfect working elevator and carrier. Fig. 96 shows a section of an elevator. The chain runs over and is operated by a sprocket gear at the head with fast and loose pulleys. The scrapers are of wood,

3 inches wide and  $11\frac{1}{2}$  inches long, bolted to lugs or projections on the chain. When run at from 50 to 70 revolutions per minute it will elevate from 5 to 10 bushels per minute. It works at an inclination or carries on the level.

Fig. 97 shows the arrangement of a plant for making cider on a large scale, as described by Paul Hassack.\* The apples are shoveled direct from the vehicle in which they are brought to the plant into the shed *A*, which is divided into not too large compartments. It is not advisable to pile the apples more than 3 to  $4\frac{1}{2}$  feet high, as otherwise, when the weather is

FIG. 97.



unfavorable, the entire pile may become heated, and rotting, browning or the formation of acetic acid set in. Alongside the shed runs up to, underneath the roof of the press-room the elevator *B*, which conveys the apples to the grinder *C*, the finely ground pulp falling into the receptacle *D*. The latter is furnished with a wooden tube *E* which can be closed, and leads to the press-room. By opening a slide a quantity of pulp just sufficient for one layer is allowed to run from the

\* "Gärungs-Essig."

tube. *F* is a press-platform equipped with wheels and running on a track. Upon this the pulp is uniformly spread, layer upon layer, each layer enclosed in a press cloth and a rack between each layer. According to the size of the press eight to twelve such layers are made into a cheese. The platform is then pushed under the press *G*, which is put in action by the motor *M*. In the commencement of the operation pressing has to be done carefully and not too suddenly to avoid bursting the press cloths, a more powerful pressure being applied only towards the end of the process when the juice runs off more slowly. In the meanwhile the next cheese is prepared.

*Testing the Must as to its Content of Acid and Sugar.*—With the exception of the grape but few varieties of fruit contain acid and sugar in such proportions and in such quantity (generally too much acid and too little sugar) in that the must obtained from them will yield, when subjected to fermentation, a drinkable and durable wine. Wine whose content of acid exceeds 1 per cent. is too sour to the taste, and one containing less than 5 per cent. of alcohol cannot be kept for any length of time. Now as all fruit wines may be called artificial wines, and a natural product has consequently to be improved in order to make it more agreeable and wholesome, it is necessary to find ways and means by which the object can be accomplished in a manner most conformable to nature. For this purpose a knowledge of the content of acid and sugar in the fruit-must is required.

To find the quantity of acid, compound a determined quantity, about 50 cubic centimetres, of must with about 5 grammes of purified animal charcoal,\* boil the mixture about five minutes, and after cooling replace the exact quantity of water lost by evaporation. After shaking bring the whole upon a

\*Bone-black which is first boiled with solution of sodium carbonate for some time, and then after washing and extracting with hydrochloric acid is again washed and dried.



coarse paper-filter in a glass funnel, and let it run off. Of the clear and generally colorless filtrate bring 6.7 cubic centimeters into a small beaker, add sufficient distilled water to form a layer of fluid 2 to 3 centimeters deep, and color red with 5 to 10 drops of litmus tincture. While holding the beaker in the left hand and constantly moving it slowly in a horizontal direction, allow to run or drop in from a pipette, graduated in  $\frac{1}{10}$  cubic centimeters and filled to the O-mark, decinormal liquid ammonia until the last drop no longer changes the color of the fluid, and the place where the drop falls appears as if made clear by a drop of water. Now prevent a further flow of the ammonia by closing the pipette with the index finger of the right hand, and read off the quantity of ammonia consumed. The must examined contains as many thousandths of malic acid as cubic centimeters of liquid ammonia were required to color the fluid blue.

Now if the examination shows that a must contains more than 8 parts of acid per thousand, it is evidently too sour for the preparation of a palatable and wholesome fruit wine, and hence must be diluted to such a degree as to reduce the content of acid to 6 or at the utmost to 8 parts per thousand. The calculation for this dilution is very simple, and consists in multiplying the acid per thousand parts present by 100 and dividing with the content of acid the wine is to have, the entire volume containing the desired acid per thousand being thus obtained. If, for instance, 18 parts per thousand of acid have been found in currant-must and the wine is only to show  $6\frac{1}{2}$  parts per thousand, then  $\frac{100 \times 18}{6\frac{1}{2}} = 276.923$ , in round num-

bers = 277, *i. e.*, 277 parts by measure of water have to be added to every 100 parts by measure of must.

The content of acid in the must thus forms the initial point for the dilution in order to obtain, after fermentation, wine with a determined quantity of acid. To be sure the content of acid is sometimes increased by fermentation, some succinic acid, as previously mentioned, being formed and perhaps also

some acetic acid. Sometimes, however, the content of acid decreases, which is very likely partially due to the water used for the dilution of the must containing earthy carbonates (lime, magnesia). It is, therefore, best not to have too much acid in the must, since, if the finished wine should be lacking in acid, it can readily be remedied by a suitable addition of tartaric acid, which is, however, not the case when it contains too much free acid.

The determination of the sugar in must presents less difficulty and has already been fully described, hence there remains only the question how much sugar has to be added to the must in order to obtain a durable wine.

Numerous analyses have shown that there is scarcely any grape wine which contains less than 7 per cent. by weight of alcohol, while in more generous wines the content rises to 12 per cent. and more. Fruit-wines in order to possess good keeping properties should never show less than 7 per cent. by weight of alcohol, but there is no reason why they should not contain as much as 10 per cent. The advantage of the latter content is evident, the wines being thereby almost absolutely protected from spoiling, while they improve in aroma and taste, the various kinds of ether being only formed in wine rich in alcohol.

The manner of calculating the quantity of sugar which has to be added to the must to give the wine the desired content of alcohol, will be best shown by the following example: Suppose 135 liters of must which contains 4 per cent. of sugar are to be changed into must with 15 per cent. of sugar.

For this purpose deduct from the weight of the must (which for the sake of simplicity we will consider equal to its volume) the weight of the sugar contained therein, multiply by the difference the per cent. of sugar the must is to contain, divide the product by 100 less the per cent. of sugar, and deduct from the quotient the per cent. of sugar already present in the must. For instance: 135 liters of must with 4 per cent. of sugar are to be changed into must with 15 per cent. of sugar.

In 135 liters are contained 6.4 kilogrammes of sugar;  $135 - 5.4 = 129.6$ , which multiplied by 15 = 1944; this number divided by  $100 - 15 = 85$  gives 22.87. Deduct from this 5.4, and there remain 17.47 kilogrammes of sugar which have to be added to the must to give it 15 per cent. of sugar.

For 325 liters of must with  $3\frac{1}{2}$  per cent. of sugar to be changed into must with 20 per cent. of sugar the calculation would be as follows:

$$\frac{(325 - 11.375)20}{100 - 20} = \frac{313.625 \times 20}{80} =$$

$$\frac{313.625}{4} = 78.406 - 11.375 = 67.03 \text{ kilogrammes of sugar to be added.}$$

600 liters of must with 6 per cent. of sugar are to be changed into must with 22 per cent. of sugar:  $\frac{544 \times 11}{39} - 36 = 117.4$  kilogrammes of sugar.

The above examples will suffice to enable any one to execute the calculations as required.

The above calculations are based upon pure, anhydrous grape sugar, an article which does not exist in commerce, and hence has to be replaced either by commercial grape-sugar (glucose) or cane-sugar. Glucose, however, containing as a rule only 67 per cent. of anhydrous grape-sugar,  $1\frac{1}{2}$  times the quantity calculated above must be used, thus in the last example 176 kilogrammes instead of 117.4. With cane-sugar the proportion is the reverse, 171 parts by weight of cane-sugar being equal to 180 parts by weight of anhydrous grape-sugar; hence the per cent. of anhydrous grape-sugar calculated according to the above method must be multiplied by the fraction  $\frac{171}{180}$  or the factor 0.95. According to this, instead of the 117.4 kilogrammes of grape-sugar in the last example, 111.73 kilogrammes of cane-sugar will have to be used.

*Glucose.*—Pure glucose being identical with the sugar in sweet fruits, its use for sweetening fruit-juices intended for the



preparation of wine is perfectly justifiable. With the dispute still carried on with honest weapons, whether it is permissible to assist nature with glucose when it fails to succeed in its labor of forming sugar in abundance, we have here nothing to do, since we know that the principal product—alcohol or spirits of wine—and almost the only one which passes into the wine by the fermentation of sugar, possesses the same properties whether it be formed from fruit-sugar or from glucose, and that neither one or the other can be injurious to health in the state of dilution in which it presents itself in the wine, provided the latter be used in moderation. The must might be sweetened, as is frequently done, with cane-sugar which occurs in sugar-cane, in beet root, in sugar-maple, etc. But with the use of glucose we are one step in advance, since cane-sugar before fermenting is first resolved into a mixture of dextrose (glucose) and levulose. Commercial glucose is never pure, as it contains, besides about 15 per cent. of water, of which about 6 per cent. is water of crystallization, about 18 per cent. of dextrin or similar substances, and some gypsum. It has a white color, and is found in commerce packed in boxes into which it is poured while in a fluid state and gradually congeals to a hard mass. It is odorless and has a faint sweet taste. On heating it becomes smeary and, finally melts to a yellowish syrup. Its content of anhydrous fruit-sugar varies between 62 and 67 per cent. Inferior qualities contain either less sugar, or have a more or less dark color, and a disagreeable odor and taste. Independently of the content of sugar, glucose to be suitable for the preparation of wine, should show no odor or by-taste.

The accurate determination of the content of pure sugar in glucose is connected with some difficulty. But few manufacturers are provided with the necessary materials for making the analysis with Fehling's solution, and besides a certain amount of skill is required for obtaining accurate results by chemical tests. In consideration of this, Anthon of Prague has devised tables which are based upon the varying specific gravity of different saturated solutions of glucose, or rather

upon its solubility in water. While 1 part of anhydrous grape-sugar requires for its solution 1.224 parts of water at 53.6 F., the foreign admixtures accompanying it dissolve in every proportion in water. Hence a saturated solution of glucose will show a greater specific gravity the more foreign substances it contains. In Anthon's tables is found the specific gravity and from this the content of anhydrous grape-sugar or glucose in the solution. In preparing a solution of starch-sugar for examination care must be had that it is completely saturated. Heat must not be used for effecting the solution, but a certain quantity of the glucose to be examined is rubbed in a mortar with one-half its weight of water at 53.6° F., and after pouring the thickish, turbid fluid into a tall beaker it is allowed to stand until clear. Anthon's table is as follows:—

Specific gravity of the solution saturated at 53.6° F.	Contains of foreign substances.	Specific gravity of the solution saturated at 53.6° F.	Contains of foreign substances.
1.2066	0 per cent.	1.2522	25 per cent.
1.2115	2.5 “	1.2555	27.5 “
1.2169	5.0 “	1.2587	30.0 “
1.2218	7.5 “	1.2631	32.5 “
1.2267	10.0 “	1.2665	35.0 “
1.2309	12.5 “	1.2703	37.5 “
1.2350	15.0 “	1.2740	40.0 “
1.2395	17.5 “	1.2778	42.5 “
1.2439	20.0 “	1.2815	45.0 “
1.2481	22.5 “		

*Cider from Apples.*—The expressed juice from well-selected apples, properly prepared, forms a lively, sparkling liquor far superior to many wines. It is quite a favorite article of home production, nearly every farmer in regions where apples are grown, making his barrel of cider for use through the winter, but a large amount finds its way into the city markets. A considerable quantity is also consumed in the shape of bottled cider, “champagne cider,” “sparkling cider,” and similar substitutes for, or imitations of, champagne wines.

In England and France considerable quantities of cider find their way into the markets, though it is there, as here, largely an article of home consumption. Certain parts of those countries are famous for the quality of their ciders, notably Normandy, in France, and Herefordshire and Devonshire, in England.

The Municipal Laboratory of Paris deduces from analyses of pure ciders from different parts of France the following as a type of composition for pure ciders :

Alcohol, per cent. by volume . . . . .	5.66
Extract. per liter, at 212° F. . . . .	30.00
Ash . . . . .	2.80

Other analyses of pure ciders, from different parts of France, published by M. G. Lechtartier, have shown great variations from this type, and show the necessity for the examination of large numbers of samples from various parts of the country for the establishment of a proper standard of analysis.

*Analyses of Ciders by the United States Agricultural Department.*—The samples for the investigation were purchased in the city in the same manner as samples of wine and beer :—



Designation.	Serial No.	No. of analysis.	Specific gravity.	Alcohol by weight.	Alcohol by volume.	Total solids.	Free acids, as malic.	Sugar, as dextrose.	Ash.	Albuminoids.	Carbonic acid.	Polarization cane-sugar scale.
<i>Well-fermented ciders.</i>												
Draft cider ("extra dry") ...	4830	1	1.0132	p. ct. 4.18	p. ct. 5.23	p. ct. 3.31	p. ct. .602	p. ct. (1)	p. ct. .396	p. ct. .038	p. ct. —	0 —19.5
Bottled cider, known to be pure .....	4832	2	1.0003	8.09	10.05	1.88	.456	—	.279	.063	trace	— 7.0
Bottled cider .....	4833	3	1.0007	6.28	7.83	1.80	.376	—	.340	.044	—	— 6.1
Bottled "extra dry russet" cider .....	4834	4	1.0264	4.48	5.61	5.52	.339	—	.393	.051	—	—35.2
"Champagne cider," bottled.	4835	5	1.0223	4.08	5.10	5.02	.567	—	.310	.050	.161	—23.4
"Champagne cider," bottled.	4836	6	1.5143	5.45	6.79	3.69	.361	—	.415	.038	.120	—20.4
"Sparkling cider," bottled. .	4927	7	1.0306	3.63	4.54	5.92	.113	—	.506	—	(2)	—33 8
Average .....	—	—	1.0154	5.17	6.45	3.88	.402	—	.377	.044	—	—
<i>"Sweet" or incompletely fermented ciders.</i>												
Draft cider .....	4829	1	1.0587	0.65	0.81	9.34	.565	—	.315	.069	—	—41.6
"Sweet" cider .....	4831	2	1.0516	0.61	0.77	9.59	.302	—	.270	.063	—	—34.2
"Sweet" cider (draft) .....	4837	3	1.0567	0.20	0.25	9.53	.575	—	.283	.075	—	—48.4
Do .....	4838	4	1.0203	3.46	4.33	3.84	.302	—	.374	.044	—	—24.2
Do .....	4839	5	1.0552	0.55	0.67	9.75	.409	—	.336	.031	—	—48.5
Do .....	4841	6	1.0355	2.96	3.71	6.98	.478	—	.348	.069	—	—39.1
Average .....	—	—	1.0455	1.40	1.76	8.17	.405	—	.321	.059	—	—

<sup>1</sup> A circumstance arising after the samples had been thrown away seemed to throw considerable doubt upon the determinations of sugar, which were made by an assistant, and the entire set had to be thrown out.

<sup>2</sup> Determinations of the carbonic acid in three different bottles gave the following results: .728, .654, .482.

The choice of the varieties of apples is of great importance in the manufacture of cider. All apple juice will not make equally good cider, even if it is equally well handled. It is not always the best flavored apple or the best tasting juice that will make the best cider. Indeed, as a rule, the best cider is made from apples which are inferior for table use, such as the crab-apple and the russet. But it is a pretty general rule that the most astringent apple will make the best cider. This astrin-

gency is due to an excess of tannin. While a portion of this tannin is changed to sweetness, a considerable portion remains, which serves to render the cider more easily and thoroughly clarified and to make it keep better. The tongue alone being, however, not sufficient to detect the tannin in apples, the following will serve as a reliable test: Express the juice of a few apples and add a few drops of isinglass, which combines with the tannin and forms a precipitate. From the greater or smaller quantity of this precipitate a conclusion can be drawn as to the quantity of tannin present. The specific gravity of the juice, which may vary between 1.05 and 1.08 should be determined. The greater the specific gravity of the juice the better the respective variety of apple is for making cider. According to these directions, the raw material should be selected, though in most cases it will be necessary to use a mixture of different varieties. In France, for a quality of cider which will keep well, the apples are mixed in the following proportions:  $\frac{2}{3}$  bitter-sweet and  $\frac{1}{3}$  sweet apples. If a sweet cider is wanted not intended to be kept for a long while,  $\frac{1}{3}$  bitter-sweet and  $\frac{2}{3}$  sweet apples are used.

The apple, like every other fruit, consists of solid and fluid constituents. The solid constituents are the skin, core, seeds, as well as the pulp in the cells of which the fluid constituents—the juice—are enclosed. The solid insoluble constituents consist chiefly of cellulose, albuminous substances, pectose, mucilage and other less insoluble substances. The average proportion between solid and insoluble substances and juice is of course subject to wide fluctuations, according to the nature of the soil, season of the year and degree of ripeness.

Generally speaking, the composition of the apple may on an average be given as follows:

Solid substance (pulp)	.	.	3 to 7 per cent.
Juice	.	.	93 to 97 per cent.

The juice constituents contain about :

Water . . . . .	80 to 88 per cent.
Sugar . . . . .	9 to 18, even up to 24 per cent.
Acid . . . . .	0.6 to 1.8 per cent.
Extractive substance . . . . .	1.3 to 3 per cent.

The juice pressed from apples is called must or cider. The sugar in the must is a mixture of different kinds of sugar varying greatly in proportion, and consists of dextrose, laevulose and sucrose. The acid of the apple, as well as that of the pear, consists of malic acid, and frequently also of small fractions of citric acid ; tartaric acid, however, is never present. Must with less than 5 per cent. of acid has an insipid taste, and consequently an addition of artificial malic or citric acid has to be made to musts augmented with water in order to improve the taste. On the other hand, when the must contains too much acid, the latter cannot be fixed with calcium or potassium carbonate, but should be reduced by the addition of water and sweetening with sugar. The extractive substances of apple-must consist of tannin 0.2 to 0.6 per cent., pectin bodies 4 to 4.5 per cent., albuminous substances and mucilage, various soluble mineral substances and a series of gums thus far undetermined.

The apples intended for the preparation of cider should be allowed to attain complete maturity, which is recognized by their color, the dark hue of the pips, little specks covering the skin, and by the sharp and agreeable ethereal odor emanating from them. In fact they should be allowed to remain on the trees as long as vegetation is active or until frosts are apprehended, for thus the conversion of the starch into sugar is best effected and their keeping better secured than by storing. They should be gathered by the hand to prevent bruising and coming in contact with dirt. They are then placed in piles and allowed to sweat. This sweating process has a tendency to ripen the fruit and make it uniform, thereby improving the flavor as well as the quality and strength of the cider in consequence



of the apples having parted with six or eight per cent. of water. The strongest cider is made from apples containing the smallest percentage of juice, and, in its aqueous solution, the largest proportion of saccharine matter. If the weather be fine, the piles may be exposed in the open air upon clean sod or where this is wanting upon boards or linen cloths, but under no circumstances should the apples be placed upon the bare ground or upon straw, as they contract an earthy or musty taste which is afterwards found in the cider.

After sweating and before being ground the apples should be wiped with a cloth to free them from exudation and adhering particles of dirt, and if any are found bruised or rotten they should be thrown out. Ripe, sound fruit is the only basis for a good article of cider, and the practice of mixing rotten apples with the sound, as is frequently done and even advocated by some, cannot be too strongly condemned. Mellow or decaying apples have lost almost all their perfume, a certain quantity of water by evaporation, and a large portion of their sugar. Rotten apples yield a watery liquid of an abominable taste, which prevents the cider from clarifying and accelerates its acetification.

The apples being wiped, sorted, and, if necessary, mixed in the desired proportions, are now brought into the grinder and reduced to an impalpable pulp. By this operation the numerous infinitesimal cells of the apple should be thoroughly broken up so as to permit the free escape of the juice when under pressure, and the machine which accomplishes this most effectually is the best for the purpose. If the cells are not thoroughly torn asunder, their tendency is to restrain and hold, as it were, in a sack much that otherwise would escape. As regards the crushing of the seeds there is a diversity of opinion, some holding that they communicate to the cider a disagreeable bitterness and acidity, while others consider them as rendering the cider more alcoholic and making it keep better.

According to M. Bergot, for cider of superior quality it is

preferable not to crush the seeds, because the diffused odor of the essential oil would undoubtedly injure the fine taste of certain notable products. For ordinary cider the crushing of the seeds will, on the other hand, be of advantage, because their essential oil helps to give to the cider the bouquet which it otherwise lacks. For cider intended to be converted into brandy the seeds must, however, be crushed. The grinder should be cleansed with hot water every evening, or at least every third day.

The treatment to which the pulp obtained by grinding is subjected varies according to the color the cider is to have. Where the consumer prefers a pale-yellow color the pulp must at once be pressed, while for a darker color it is allowed to stand 12 to 18 hours.

The next step in the operation is pressing. The various kinds of presses, racks, and manner of laying up the cheese have already been described. The primitive custom of laying the cheese was to lay upon the platform of the press a quantity of straw, upon which a quantity of pomace was placed, and the edges secured by laps of straw, thus alternating straw and pomace until the pile was complete. The object of using the straw was to hold the mass together while it was being submitted to pressure, and also to serve as a means of exit for the cider. An improvement was in the substitution of hair-cloths, and within the past few years the adoption of the cotton press-cloth and racks to hold the pomace in laying up the cheese for the press. The racks have already been described. The press-cloth is woven from yarn made expressly for the purpose and is of equal strength in warp and filling. The use of straw in laying up the cheese should be entirely discarded, as the slightest mustiness imparts an unpleasant odor to the cider.

The pressure applied to the cheese should be slow at starting and then gradually increased until finally the full force is applied. The juice as it comes from the press runs through a fine hair-sieve into a receiver. With a good press about 65 to 75 per cent. of juice will be obtained.

After the cider has been extracted and the cheese removed from the press the pomace may be utilized for the manufacture of vinegar, as previously described. In France it is, however, used for the manufacture of the small cider. The method is as follows:—After the extraction of the pure cider by the first pressing, the pomace is taken from the press, and after adding 12 litres of water for every hectoliter of apples used, the mass is allowed to macerate 15 to 20 hours, care being had to stir every two or three hours. Then this pulp is put a second time under pressure and a quantity of juice extracted equivalent to the amount of water added.

*Extraction of the juice by diffusion.*—Diffusion, which gives such excellent results in the extraction of sugar-beets, has also been applied to extract the soluble constituents of the apple. In practice this method might be suitable for persons having no cider press and only a small quantity of apples to handle. The quality of cider is nearly equal to that obtained by three pressures, and the juice obtained is almost as rich as that yielded by the press.

Successful experiments in expressing the juice of the grape by means of the centrifugal would indicate that the same method might also be applied to apples.

The freshly-expressed apple-juice is either sold as sweet cider or subjected to fermentation. Fermentation in sweet cider is retarded by pasteurizing, carbonating, or the addition of preservatives. The objections urged against pasteurizing or sterilizing fresh apple-juice are that a "cooked" taste is added to the juice, and that it is impracticable to hold the juice sterile for more than a limited period. Experiments to develop a method for sterilizing apple-juice in wooden, tin and glass containers have been made by H. C. Gore,\* and his investigations demonstrate that only a slight cooked taste is produced by the heat treatment required, and that it is a sim-

\* U. S. Department of Agriculture, Bureau of Chemistry, Bulletin No. 118. "Unfermented Apple-juice."



ple matter to protect the juice from inoculation after sterilizing. A summary of these investigations is here given :—

(1) “The experiments show conclusively that it is possible to sterilize apple-juice in wooden containers, the product remaining sound for at least six months under actual observation. The precautions which must be taken to insure this are as follows: First paraffin the containers on the outside, then sterilize, and fill with juices heated to between 149° and 158° F. (65° to 70° C.); seal, taking measures to relieve the vacuum produced by the contraction of the juice on cooling by filtering the air through cotton. Twenty-four 10-gallon kegs successfully stood a severe shipping test, showing no loss due to fermentation of the juice. The juice so prepared was found to be palatable, and acceptable as a summer drink.

(2) “It is demonstrated that apple-juice can be successfully sterilized in tin containers, using the type of tin can sealed by the mechanical process, excluding all metals from contact with the juice except the tin of the can. Where lacquered cans are used the contamination with tin was reduced about one-half. Apple juices were canned and sterilized by heating in a hot water-bath, up to the temperature of 149° F. (65° C.) for a half hour, and then allowed to cool. These juices possessed only a slight cooked taste due to the heating and retained much of their distinctive apple flavor. It was found that from finely flavored apple-juice a first-class sterile product could be made, while a poorly flavored apple-juice yielded an inferior product. The process conditions mentioned were not quite thorough enough to sterilize all of the varieties canned. A slight increase in the temperature or time of processing, or both, should be made, the temperature not to exceed 70° C. (158° F.) in any case.

(3) “The best treatment for sterilizing in glass was found to consist in heating for one hour at 149° F., or for one-half hour at 158° F. Heating for one hour at 158° did not produce marked deterioration in flavor, a half hour being allowed in all cases for the juice to obtain the temperature of the water-bath.

(4) "It was shown that the great bulk of the insoluble material naturally contained in apple-juice can be removed by means of a milk separator."

These investigations extended also to carbonating fresh apple-juice and the conclusions arrived at are as follows:

"It is possible to carbonate the juice slightly before canning or bottling, thus adding a sparkle to the product. A flavor foreign to fresh apple-juice is also added, however, and uncarbonated sterile juice will resemble fresh apple-juice more closely. Carbonating by the addition of water charged with carbon dioxid was considered by some to injure the flavor, lessening the characteristic fruit flavor by dilution. In the opinion of others a heavy, rich juice was improved both by the charge of carbon dioxid and by the consequent dilution. Experiments indicated that the danger of contamination by mold growths was lessened by maintaining an atmosphere of carbon dioxid above the surface of the juice after opening."

When apple-juice is sold in bulk a small amount of benzoate of soda is, as a rule, added to retard fermentation, one-tenth of 1 per cent. being tolerated by regulation in the United States. H. C. Gore's investigations demonstrated that benzoate of soda in quantities varying from 0.03 to 0.15 per cent., while it checks the alcoholic fermentation, allows other organisms to develop—notably the acetic acid ferment—whereby the palatability of the product as a beverage is destroyed.

H. C. Gore has also investigated the cold storage of apple cider,\* and the summary of the results of these investigations is here given:

(1) "Ciders prepared from apples free from decay chilled rapidly to the freezing-point immediately after pressing, and then held in cold storage at 0° C. (32° F.) remained without noticeable fermentation for a period of from thirty-six to fifty-seven days, an average of fifty days for the Tolman, Winesap,

\* U. S. Department of Agriculture, Bureau of Chemistry, Circular No. 48, "The Cold-Storage Apple Cider."

Yellow Newtown, Ralls, Gilpin, and Baldwin varieties, and of eighty-three days in the case of the Golden Russet, Roxbury Russet, and Kentucky Red.

(2) "These ciders were held for a period of from ninety to one hundred and nineteen days, an average of ninety-nine days for the first six varieties and of one hundred and twenty-five days for the last three, before they fermented sufficiently to be considered as becoming "hard" or "sour."

(3) "The ciders were found to have suffered no deterioration (with the exception of the Tolman), but rather had become more palatable during storage."

The apple-juice to be fermented should be tested with a must-spindle or densimeter as to its content of sugar. A good quality of juice will generally range from 10 to 14 per cent. If less than 10 per cent. the juice will not make a cider that will keep, though, if the flavor in other respects is all right, a beverage for immediate use may be produced from it.

When the juice has been tested and, if found wanting in saccharine strength, corrected by the method previously given, the next step in the operation is fermentation. For this purpose the juice is brought into casks. Regarding the size of the latter it may be said that, as a rule, the juice ferments more uniformly and more steadily, and retains the carbonic acid better with the use of larger casks, though it develops somewhat more slowly than in smaller containers. For the production on a large scale of cider of first-rate quality the use of large casks can, therefore, be recommended. However, if the cider is to be used for daily consumption, and perhaps be directly drawn from the yeast, smaller containers are preferable, there being less danger of the cider becoming mouldy or sour. The casks should be scrupulously clean, and new ones must be freed by steaming or washing with hot water from all extractive substances, otherwise the cider will acquire a disagreeable taste and dark color.

In many places the fermentation-casks are filled by means of a power pump which delivers the juice to the receptacles



placed in adjacent rooms or in another building. When the press-room is over the fermentation room, filling is accomplished by gravity. Hose-pipes are largely used for this work, but brass or copper must be used for all metal fittings. The less the juice comes in contact with the air after it leaves the press the less liable it is to be contaminated with various undesirable organisms. The pumps and pipes must be kept scrupulously clean.

In Fig. 97 p. 386 the juice running off from the press through the pipe *H* is freed from the principal particles of pulp in the box *J*, which is fitted with two or three wire-sieves of different fineness. This box is located above the collecting vat *K*.

The fresh juice having been brought into the casks, fermentation is still left in many places to the organisms normally present on the fruit and those which may at the time of grinding and pressing enter the juice from contact with the air, the machinery and the vessels. Fermentation in this case does not always progress as normally and favorably as required for the production of a sound, palatable and durable cider. The various races of yeast present on the apple possess but little fermenting power and the elliptic wine yeast (*saccharomyces ellipsoidus*) which has to be taken chiefly into account, being generally represented only in very small quantities, is stifled and readily suppressed. It is a well-known fact that, generally speaking, apple juice ferments completely only with difficulty. This appears to be due to the fact that the natural cane sugar, which is frequently present in considerable quantities in apple and pear juices, is fermented with great difficulty by the organism normally present on the fruit, and to assure the ascendancy of the true yeasts and thus give them the control of the entire process of fermentation, the practice of sowing the juices with pure cultures of yeast has been introduced. In Germany all the important factories employ these cultures, which are obtained in small flasks from the Royal Pomological School at Geisenheim. With the use of pure

culture of yeast it is best to add it to a smaller quantity of sterile juice previously heated to 140° F. and then cooled to 68° F. When fermentation in the sterile fluid is most vigorously developed it is added to the juice to be fermented.

Another cause of the difficult fermentation of apple juice is the frequent want of nitrogenous combinations required for the nutriment and propagation of the yeast. This may be remedied by the addition of 20 grammes of ammonium tartrate or ammonium chloride per hectoliter of juice; in place of it the same quantity by weight of ammonium phosphate or ammonium carbonate may also be used. Such an addition should also be made with the use of pure culture of yeast.

The first or tumultuous fermentation of the apple juice is in some places effected in open vats, this method being generally preferred with juice not previously strained, so as to be able to remove during fermentation the insoluble constituents of the must which are forced to the surface.

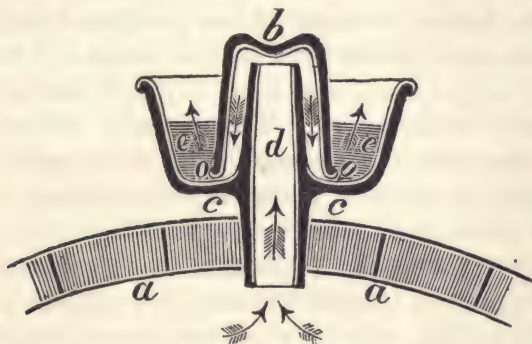
When, however, the must has been purified by straining or filtering over thoroughly washed sand, fermenting in casks is preferable. The casks are filled about three-quarters full and equipped with a ventilating bung to prevent the entrance of germ-laden air. There are various constructions of ventilating bungs but the principle is in all cases the same, namely, to allow the escape of the excess of carbonic acid and prevent the entrance of air. The best protection of the must is the carbonic acid developed during fermentation, because, on the one hand, neither mould or acetic acid formation can appear for want of oxygen, and, on the other, the excitors of these decompositions cannot develop in an atmosphere of carbonic acid. Care should, therefore, be taken that until fermentation is finished and the cask has been entirely filled and bunged, an atmosphere of carbonic acid always lies over the cider in the empty space of the cask.

The fermentation funnel or ventilating funnel, Fig. 98 which is largely used in Germany, is a simple device for controlling the air. It is generally made of pottery or porcelain, though

it can also be constructed of metal, for instance, aluminium. It consists of two parts, the actual funnel *c* with the tapering pipe *d*, which is secured air-tight in the bung-hole and is filled half-full with water, and a cup-like vessel *b*, which is placed over the elongated portion of the pipe of the funnel. The carbonic acid escaping from the cask passes through the pipe into the cup *b*, forces back the water at *o* and escapes at *e* from the open portion of the funnel, the entrance of air being on the other hand prevented by the water.

The first or tumultuous fermentation runs its course, according to temperature and other conditions, in two to four weeks,

FIG. 98.



the temperature being under otherwise normal conditions, the most important factor. The higher it is, the more energetically fermentation sets in and the more rapidly it runs its course. While formerly a not too tumultuous course of the first fermentation was not desired by many manufacturers and the temperature was kept relatively low, most of them have now arrived at the conclusion that as energetic a course as possible of the first fermentation is the best guarantee for a good product, and the temperature for the first stage of fermentation should be at least between  $59^{\circ}$  and  $68^{\circ}$  F. When the first fermentation has run its course, which is recognized by the cessation of the hissing sound made by the carbonic



acid gas, the cider is drawn off from the sediment into clean, unsulphured casks, furnished with a ventilating bung. The casks are placed in a cellar or a cool room having a temperature of 40° to 50° F., and the cider is left to the second or after fermentation. The casks should be constantly kept full, and abrupt variations in the temperature carefully avoided and provided against. Generally speaking, the more energetically the first fermentation has run its course, the more quietly the second fermentation will progress, and *vice versa*. By the second fermentation, the remainder of the sugar is decomposed, there is but a slight evolution of carbonic acid, the yeast as well as the albuminous substances in the must settle on the bottom, and the cider becomes more or less clear.

When the second fermentation has progressed to the desired degree, the cider is drawn off into other casks and fined. According to one method this is done with isinglass, 1½ ozs. of it being allowed for each cask. This quantity is dissolved in 1 pint of cider over a moderate fire, and the solution when cold, poured with constant agitation into the cask. Drawing off may be commenced after eight days.

A better method of clarification, which at the same time increases the purity of taste of the cider, is as follows: For each barrel of 30 gallons, take 4 lbs. of fresh wheat bran, and, after washing it twice in hot water to remove all soluble substances, press out thoroughly. Now dissolve about 2 drachms of alum in a bucketful of hot water and pour the solution upon the bran. After 6 to 8 hours take the latter from the alum water and press as before. The bran is best used before the cider is racked off for the third and last time. Stir it into the cider, and then draw off the latter through a fine strainer into the actual storage barrel. The cider first passing through the strainer is generally somewhat turbid, and must be poured back until it runs off clear.

In France, the cider is generally clarified by dissolving 2 ozs. of catechu in 1 quart of cider and adding the solution to 100 quarts of cider, with constant stirring. The tannin thus added

precipitates the albuminous matters, the result being a clear cider which will not blacken in the air.

It is always advisable before fining large quantities of cider to make a clarifying experiment on a small scale, the content of tannin in the fluid being frequently so small that the clarifying agent added is ineffectual. In such cases a small addition of tannin in the form of an alcoholic-aqueous solution previous to the addition of the clarifying agent can be recommended. However, as a perfectly bright product is not always obtained by fining, filtering will have to be resorted to. Filters of various types, such as bag filters, cellulose filters and asbestos filters are in use for this purpose. Filtering cider appears to be a process much more difficult than filtering wine made from grapes and should be avoided if possible. The reason for this is the presence of mucilaginous substances in the liquor.

Pared apples, if used for the production of cider, yield a product poor in aroma. Washing the apples in washing machines of special construction previous to grinding and pressing is of great advantage for the production of fine must and cider. Fairly good products can be obtained from dried American apples, the cheapest brands (waste, parings, cores) of which can be used for the purpose.

Cider intended for export must be made somewhat richer in alcohol, which is generally done by adding sufficient French brandy to increase its content of alcohol 2 per cent. Sometimes, also,  $\frac{1}{4}$  lb. of sugar for every 2 quarts of juice is added during fermentation. For shipping to tropical countries experiments might be made with salicylic acid, adding it in the same proportion as to beer, which is for beer sent in barrels  $\frac{3}{4}$  oz. for 100 quarts, and for bottled beer,  $\frac{1}{2}$  oz.

There are several methods of improving the taste of cider, but they are rather questionable, because tastes differ, and what might be considered an improvement by one would be declared a defect by another. A favorite method of improvement is as follows: For 45 gallons of cider measure off 3 quarts

of French brandy and mix it with the following substances, all finely powdered: 0.7 drachm of bitter almonds, 0.7 drachm of mace, and  $7\frac{1}{2}$  drachms of mustard-seed, and finally  $3\frac{1}{2}$  drachms of catechu, previously dissolved in water. Pour this mixture into the cider and shake the barrel frequently during the next 14 days. Then allow it to rest three or four months, and should it then not run off clear when tapped, clarify it with  $1\frac{1}{4}$  oz. of isinglass or the whites of a dozen eggs. If the color of the cider is to remain pale yellow, catechu cannot be used, and instead of isinglass or white of egg, skimmed milk is to be used for clarification. For a reddish color, which is sometimes desired, use  $1\frac{3}{4}$  drachms of powdered cochineal in place of the catechu.

Sometimes cider is prepared in the same manner as other fruit-wines. In this case  $\frac{1}{4}$  lb. of sugar is added to every quart of juice, and the latter is allowed to completely ferment in the same manner as grape wine. According to another direction, add to every 2 quarts of juice, 2 lbs. of white sugar, and boil as long as scum is formed; then strain through a fine hair-sieve and allow to cool. Now add a small quantity of yeast, stir thoroughly, let the whole ferment three weeks, and after clarifying rack off into bottles.

*Red apple wine*, or, as it is frequently called, *red wine from cider*, is prepared as follows: Boil for 2 hours 50 quarts of apple juice, 27 lbs. of honey, 1 oz. of tartar, 6 lbs. of comminuted red beets, and 3 lbs. of brown sugar. Let the fluid completely ferment, and if no apple juice is on hand to fill up the barrel during this process, use solution of sugar. When fermentation is finished, pour a mixture of 1 quart of French brandy and about 1 drachm each of pulverized cinnamon and ginger into the barrel. After three months clarify the wine and rack off.

In his treatise on "Cider," Dr. Denis-Dumont gives the following directions for bottling cider: The cider is to be bottled at three distinct periods. It should never be bottled before the tumultuous stage of fermentation is entirely completed and the liquid clarified.



*First period.* At the termination of the tumultuous fermentation, the cider still contains considerable sugar. Fermentation continues in the bottle and produces in a few weeks a large quantity of carbonic acid. In order to prevent the bottles from being broken by the pressure, champagne bottles should be selected, and care taken to have them stand upright until the development is considerably reduced. The bottles are then laid on their side, as otherwise the cider would cease to be sparkling. This cider has to be kept for a number of years, it being good to drink only when old.

*Second period,* when fermentation is more advanced, about six weeks or two months after the first period. Mineral water bottles are strong enough to hold this cider, it liberating less carbonic acid than the preceding. The bottles are left in an upright position for a few weeks only. This cider has a good flavor and is fit to drink much sooner than the preceding. It keeps for a long time.

*Third period,* when fermentation is complete or almost so, any quality of bottles may be used, a great deal less of carbonic acid being developed than in the preceding cases. The bottles should be laid down immediately after filling, in order to retain the carbonic acid which will still be developed. This cider is not sparkling; it is, however, lively, strong, and has a fine flavor.

The bottles should, in every instance, be well corked, and the corks, for the sake of safety, tied. The cider is very good when kept in small bottles, better in quart bottles, and best in jars holding two quarts. A few moments before opening a bottle of sparkling cider, it is advisable to provide a minute opening for the escape of the gas by piercing the cork with a fine punch. As soon as the tension of the gas has become sufficiently weak, the cork is allowed to blow out in the same manner as with champagne. Without this precaution, most of the cider might be thrown up to the ceiling.

In the island of Jersey, where the manufacture of cider is carried on in a very rational manner, the juice as it comes from

the press is allowed to ferment in large open vats placed in a cellar having a uniform temperature of from 53° to 59° F. On account of the large surface presented to the air, tumultuous fermentation soon sets in, and in about four or five days, or at the utmost a week, fermentation is over. The liquid is then drawn off in barrels, thoroughly cleansed and sulphured, in which fermentation continues slowly. These barrels are not entirely filled, and when the development of carbonic gas has proceeded so far that the flame of a lighted candle introduced by the bung-hole is extinguished, the liquid is drawn off into other barrels sulphured like the first. This transfer from one set of barrels to another is continued until no escape of gas is perceptible, *i. e.*, until fermentation is quite complete.

Prepared in this manner the cider will keep perfectly good for several years, and stand transportation by sea without any difficulty.

*Devonshire cider* is made from a mixture of one-third of bitter-sweet apples with a mild sour. These being gathered when thoroughly ripe are allowed to undergo the sweating process before grinding. The cider is then pressed in the usual manner and strained through a hair-sieve into hogsheads, where it remains for two or three days previous to fermenting. It is then drawn off into clean casks to stop the fermentation, but if this is very strong only two or three gallons are first put in, and after burning cotton or linen rags saturated with sulphur in the cask, thoroughly agitated. This completely stops fermentation in that quantity and usually checks it in the other portion with which the cask is then filled up. In a few weeks the cider becomes very fine. If this be not satisfactorily accomplished by the first operation, it is repeated until fermentation is completely checked and the cider is in a quiet state and in a proper condition for drinking and bottling.

*Champagne-cider*.—The manufacture of this beverage has become quite important—it resembling the ordinary but more expensive champagne-wine, and being frequently sold as such. Since the devastation of the vineyards by the phylloxera, a

large trade in the spurious champagne-wine is carried on in France. This champagne-cider if sold under its right name is an excellent beverage. It is prepared as follows: To 50 gallons of apple-juice add 12 quarts of brandy and 14 lbs. of sugar or honey. Mix the whole thoroughly, and allow it to ferment for one month in a cool place. Then add about 4 drachms of orange-blossom water, and clarify with 2 quarts of skimmed milk. The champagne is now ready and is racked off into bottles, into each of which a small piece of white sugar is thrown, and the corks of which are wired. The duration of fermentation has been stated as one month. It may, however, last a few days more or less, it being entirely a matter of observation when the most suitable time for racking off has arrived. No more rising of bubbles of gas should be observed, but fermentation must not be completely finished.

According to another process, 40 quarts of fermented apple-juice are mixed with 2 quarts of solution of sugar,  $\frac{1}{2}$  quart of rectified alcohol and 2 ozs. and 4 drachms of pulverized tartar. The mixture is allowed to stand 24 hours and then racked off into bottles, each bottle receiving a drachm of bicarbonate of soda. Cork and wire.

Another process consists in bringing into a vat 40 quarts of apple-juice, 5 lbs. of white sugar,  $\frac{1}{4}$  lb. of tartar, 1 pint of rectified alcohol,  $\frac{1}{4}$  pint of yeast and 1 oz. and  $2\frac{1}{2}$  drachms of acetic ether. Shortly before fermentation is finished the mixture is drawn off into bottles, each of which has previously been provided with a small piece of sugar. Clarification with isinglass, white of egg or skimmed milk must, of course, precede the drawing off into bottles. The bottles must be thoroughly corked and wired in the same manner as genuine champagne, and laid in a cool cellar.

Cider serves frequently as a basis for artificial wines, *genuine* Burgundy, sherry or port-wine, prepared from cider mixed with suitable substances, being frequently served even in first-class hotels. Nothing could be said against these beverages if they were sold under their proper names, because they consist



of harmless substances, which cannot always be said of the genuine wines, they being only too frequently adulterated with substances injurious to health.

*Burgundy.*—Bring into a barrel 40 quarts of apple juice, 5 lbs. of bruised raisins,  $\frac{1}{4}$  lb. of tartar, 1 quart of bilberry juice and 3 lbs. of sugar. Allow the whole to ferment, filling constantly up with cider. Then clarify with isinglass, add about 1 oz. of essence of bitter almonds, and after a few weeks draw off into bottles.

*Malaga Wine.*—Apple juice, 40 quarts; crushed raisins, 10 lbs.; rectified alcohol, 2 quarts; sugar solution, 2 quarts; elderberry flowers, 1 quart; acetic ether, 1 oz. and 2 drachms. The desired coloration is effected by the addition of bilberry or elderberry juice; otherwise the process is the same as given for Burgundy.

*Sherry Wine.*—Apple juice, 50 quarts; orange-flower water, about 2 drachms; tartar 2 ozs. and 4 drachms; rectified alcohol, 3 quarts; crushed raisins, 10 pounds; acetic ether, 1 oz. and 2 drachms. The process is the same as for Burgundy.

*Claret Wine.*—Apple juice, 50 quarts; rectified alcohol, 4 quarts; black currant juice, 2 quarts; tartar, 2 ounces and 4 drachms. Color with bilberry juice. The further process is the same as for Burgundy.

*Diseases of Cider.*—Ciders are subject to diseases which may be due to the bad quality of the apples used, a faulty method of manufacture, or bad management in the cellar.

Badly fermented cider, especially such as has merely passed through the stage of tumultuous fermentation, or has been acidified by contact with the air, is liable to produce serious disorders. The first, says Dr. E. Decaisne, being heavy and indigestible, inflates the intestines and produces diarrhœa; the second, though of a sweet taste and a piquant and agreeable flavor, does not quench the thirst, but excites the nervous system and produces flatulency; the third, which is really spoiled cider, causes inflammation of the intestines by the large amount of malic and acetic acid it contains. When in

the production of cider, water containing organic matter has been used, putrid fermentation is produced in the mass, the products of which impart some very deleterious properties to the cider.

*Acidity* in cider may be due either to an excess of malic acid or of acetic acid.

Some ciders contain too much malic acid when manufactured from apples not sufficiently ripe, or when, in mixing the apples, too large a proportion of sour apples has been taken. In both these cases the acidity may be neutralized by adding to the apple-juice 3 ounces and 8 drachms of potassium tartrate per 22 gallons. Sometimes there is an excess of acetic acid, due to the oxidation of the alcohol by long contact with the air. This defect is difficult to remedy. It might have been prevented by means of a thin coat of olive oil, as previously mentioned, or by hermetically closing the bungs. The acidity will, however, disappear by putting in the bottles a pinch of bicarbonate of soda. It must, however, be done immediately on detecting the defect.

*Viscosity or greasy appearance* of cider is recognized by the cider becoming stringy, viscous and greasy, and is due to too great an abundance of gummy substances in the fruit, a lack of tannin, and finally to defective fermentation. In order to check this malady from its first appearance, add to every 228 quarts of the cider 1 pint of alcohol or 2 grammes of catechu dissolved in 3 quarts of water. Cider may be prevented from turning viscous by the addition of sugar to the juice when it comes from the press, fermentation being thereby promoted.

The cause of cider *turning black* is an excess of oxide of iron, which, on coming in contact with air, becomes a peroxide and gives the beverage a brown color. The oxide of iron may have been introduced into the cider either by the water used in making it, or by fruit grown on ferruginous soil. By mixing such cider with 12 drachms of powdered oak bark per 22 gallons, a quantity of tannin is introduced which combines with the iron to an insoluble product that settles on the bottom of the barrel. Tartaric acid may also be used.

*Turbidity* or *lack of clarification* of cider is caused by too small a quantity of sugar in the juice, or by imperfect fermentation.

In rainy seasons the apples ripen imperfectly and contain but little sugar. Cider prepared from such fruit generally remains turbid. During seasons in which abrupt changes of temperature take place, and also when cold weather sets in very early, fermentation does not progress well, and clarification is imperfect. When the cider remains turbid after the first racking off, add a solution of 2 lbs. of sugar in 1 gallon of water to every 132 gallons of the liquid. This sugar becomes converted into alcohol and renders the cider limpid. The use of lead salt, formerly much in vogue in Normandy, is very dangerous. Persons drinking cider thus treated frequently feel sharp pains in the abdominal region, which present all the symptoms of lead colic and may even prove fatal.

An admixture of lead salt is readily recognized. Add to the suspected cider solution of potassium iodide; if lead salt be present, a yellow precipitate of iodide of lead will be formed.

*Adulteration of Cider.*—According to most of the authorities on food, cider is but little subject to adulteration. Even Hassall, who generally enumerates under each article of food a list of every conceivable adulteration that has ever been found or supposed to have been used in such food, only speaks of the addition of water, of burnt sugar as a coloring matter, and of the use of antacids for the correction of the acidity of spoiled cider. On the other hand, in France where, as previously mentioned, the consumption of cider is quite large, its adulteration is by no means uncommon. The following is considered in the Paris Municipal Laboratory as a minimum for the composition of pure cider:

Alcohol, per cent. by volume . . . . .	3.00
Extracts, in grammes per liter . . . . .	18 00
Ash . . . . .	1.7



This is for a completely fermented cider. In sweet ciders the content of sugar should exceed the limit sufficiently to make up for the deficiency of alcohol, to which it should be calculated.

In the samples of American ciders investigated by the United States Agricultural Department (see pp. 393-4), it was fully expected to find a number preserved with antiseptics. This supposition failed to be confirmed, however, for no salicylic acid was found, and in but one case was any test obtained for sulphites. None of the samples fell below the standard proposed by the French chemists, given above, and no metallic or other adulteration was discovered.

There was, however, a single exception, No. 4927 in the table of analyses, p. 394, which was an embodiment in itself of nearly all the adulterations which have been enumerated as possible in cider. It was handsomely put up in neatly-capped bottles, and was of a clear, bright color. Its tremendous "head" of gas when uncorked gave rise at once to the suspicion that it had received some addition to produce an artificial pressure of gas. The low content of free acid, together with the large amount of ash and a variable content of carbonic acid in different bottles, established the fact that bicarbonate of soda had been added, probably a varying quantity to each bottle, while the dose of sulphites added was so large that a bottle stood open in the laboratory all through the summer without souring.

*Manufacture of brandy from cider.*—Brandy is a mixture of water and alcohol produced by the distillation of a fermented liquor. It owes its aroma to the essential oil peculiar to the substance subjected to distillation.

In Normandy the heavy ciders only are distilled, *i. e.*, those containing the most alcohol.

In years when there is an abundant crop of apples, it will generally be found of advantage to distil the cider made from fallen fruit and also from early apples. The cider yielded by them does not keep well, and brings a very low price, especially when there is a large product from late apples.

Sour ciders should not be distilled, they being better utilized for the manufacture of vinegar. Spoiled cider, as a rule, makes bad brandy.

Different qualities of cider should be distilled separately. A skilled distiller can classify them by the taste, and separates them in order to obtain brandy of first and second qualities.

The cider is distilled when it is completely fermented, *i. e.*, when the largest possible quantity of sugar has been converted into alcohol. Cider from early apples generally ferments faster than that from late apples and can be distilled towards the end of December, *i. e.*, from six weeks to two months after it has been made. Cider made from late apples, during December and January, is ready for distillation three or four months later, *i. e.*, in March or April.

*Preparation of the juice for distillation.*—When there is an abundant crop of apples and barrels are scarce, the juice as it comes from the press is brought into large open vats in which fermentation progresses rapidly, but in this case some beer yeast previously mixed with a small quantity of cider is added to each vat and the temperature must be maintained between 59° and 68° F. Under these conditions the juice ferments very promptly and may be distilled eight or ten days later.

Sometimes the whole of the pulpy mass obtained by grinding the apples is submitted to distillation. In order to accelerate fermentation a small quantity of hot water containing some sugar in solution is added to the mass, also one or two thousandths of sulphuric acid, the latter regulating the progress of fermentation.

Fermentation being finished, the mass is subjected to distillation. In order to prevent this mass from adhering to the still and scorching, distillation must be conducted as slowly as possible and a small quantity of straw placed upon the bottom of the still, or, better, a piece of cloth to prevent direct contact of the mass with the heating surface.

Plums, damsons, etc., are also subjected to distillation and produce good brandy. They ferment more slowly than wild

cherries which produce the well-known cherry-bounce. Attention may here be called to the distillation of wild plums, which should be gathered in the fall when the leaves begin to drop. Some connoisseurs consider brandy made from plums equal to that from cherries. On a farm, no fruit containing sugar should go to waste, as it can be converted either into brandy or vinegar.

*Distillation.*—For distilling cider on a small scale no expensive apparatus is necessary, an ordinary still answering all requirements. Cider is distilled like wine. The still is filled about  $\frac{3}{4}$  full and after placing the head in position the joints are carefully luted by pasting strips of cloth or even paper over them. The tub holding the worm is filled with cold water and the fire started. The vapors escaping from the boiling liquid condense in the worm and run into the receiver. Heating should be done slowly, in order to vaporize as little water as possible, and especially to avoid sudden ebullition, as the boiling liquid, getting into the head, would pass through the worm and become mixed with the liquor already distilled. In such an event it would be necessary to begin distillation anew. The operation is continued until the liquid produced contains hardly any alcohol, which can be ascertained by the use of the alcoholometer or by the taste. It is unnecessary to say that care must be had to constantly renew and keep cold the water in the tub holding the worm.

Distillation being finished, the boiler is emptied, and after thorough cleansing is refilled for a second operation.

The liquid produced by successive distillations is mixed together and brought into the still a second time, whereby a liquor richer in alcohol and of a better taste is produced. It would be desirable if this second distillation or rectification could be effected by means of steam. This would prevent the empyreumatic taste which is often noticed in apple-brandy. The first and last runs of the still being of inferior quality are collected separately and poured back into the still when refilling for the next operation.

Calculations have been made to establish by means of figures



the immense advantage offered in a financial point of view by the distillation of cider. These theoretical calculations, however, are frequently very deceptive. If, on the one hand, the producer knows the content of alcohol of his cider and, on the other, the market value of the alcohol and of the cider, it will be easy for him to decide which product will pay him best.

*Pear-cider.*—The manufacture of pear-cider is very limited, and no great future can be promised for it, as even when most carefully prepared it is far inferior to apple-cider and other fruit-wines. Its preparation is best understood in England, and how little it is appreciated there is shown by the fact that three-fourths of the quantity manufactured is consumed by the farm-laborers. But any one who has large pear crops at his disposal and wishes to use a portion of them for the manufacture of a beverage should add to the pear-must one-quarter its quantity of must of bitter-sweet apples or a few quarts of black currant juice, which will improve the taste of the cider and its keeping qualities. The mode of preparation is the same as for apple-cider, though still greater care must be exercised in the choice of the raw material. The pears must have a sufficient content of sugar, as otherwise the cider would not be sufficiently rich in alcohol and at the same time they must contain a bitter substance to prevent the cider from turning sour as soon as the conversion of the sugar is effected. Hence the use of fine table pears for the preparation of cider would be simply a waste of material. The only varieties suitable for the purpose are those which when eaten from the tree produce a long-continued sharp heat in the throat and lie half a day undigested in the stomach, which, however, become sweet by long storing and lose enough of their acerbity to be no longer disagreeable to the palate. In England, the wild pear grown in hedges is generally used for the purpose. They must be ripe, but not soft or mellow.

In the northern part of France pear-must is sometimes used for the preparation of "port wine," the taste of which is very much praised. The process consists in heating 50 lbs. of must

to between 176° and 185° F. and adding 5 pounds of raisins. At this degree of heat must and raisins are brought into a barrel which is tightly bunged and placed in a cool place. When in the course of a day the must is cooled to 59° or 68° F., the raisins, which are generally put in a bag, are taken from the barrel and after bruising returned (but not inclosed in the bag) to the must, which is then allowed to ferment for 14 days. The wine is then drawn off into stone jugs which are well corked and sealed.

*Quince Wine.*—A very spicy wine can be prepared from quinces in the following simple manner: Place the quinces for a few moments in hot water and then rub them with a cloth to remove the down. Next remove the cores by means of a knife or in any suitable manner. Now pour hot water over the quinces thus prepared and boil them slowly over a moderate fire until soft. Then press out the juice and add white sugar in the proportion of 1½ lbs. to every 20 lbs. of fruits. Allow the whole to ferment in a cool room and from time to time add some sugar-water during the process. Clarification and racking off is effected in the same manner as with cider.

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## CHAPTER XXX.

### PREPARATION OF FRUIT WINES.

THE manner of obtaining the juice and appliances for that purpose have already been described in the previous chapter.

a. *From small fruits.*—One of the principal objections to wines from small fruits is that they easily turn. This can, however, be overcome by adding, after fermentation is finished, 5.64 drachms of salicylic acid to every 100 quarts. By increasing the dose to 8.46 drachms less sugar can be added to the must, which, of course, makes the beverage poorer in alcohol. A saving of sugar can be further effected without injury

to the keeping quality of the wine by a suitable mixing of juices. By working, for instance, the juices of currant, or of raspberries by themselves, a considerable addition of sugar, about 1 pound per quart, has to be made, which can, however, be reduced one-half by mixing with a juice containing some bitter principle, and later on treating the wine with salicylic acid. Thus a large field for experimenting is opened to all, and only a few hints will here be given. Raspberry-juice should be mixed with one-quarter its volume of blackberry-juice; and in the preparation of currant-wine it is especially recommended to use four-fifths of red to one-fifth of black currants, the wine obtained being far more spicy and possessing better keeping qualities. Moreover, black currants used within limits are an excellent material for improving the flavor of almost all fruit-wines. The flavor and keeping qualities of fruit-wine are also improved by throwing a couple of handfuls of crushed hazel-nuts or walnuts into the barrel, and also by the addition of 2 ounces and 8 drachms of bitter almonds, the peels of 10 lemons, 3 ounces and 5 drachms of cassia, and a few handfuls of bruised wild plums. By these means wine with a moderate content of alcohol acquires a strong taste, while its keeping quality is at the same time improved. The latter can also be effected by bringing 2 ounces and 3 drachms of tartar into the barrel during fermentation. A few other mixtures of juices may be mentioned. Blackberry-juice is better adapted to ferment by itself than any other juice from small fruits, but by the addition of  $\frac{1}{8}$  to  $\frac{1}{4}$  its weight or its volume of strawberry-juice the aroma of the wine is greatly improved. Strawberry-juice is least suitable for fermentation by itself, and should be mixed with must containing a bitter principle. The addition of  $\frac{1}{6}$  of the volume of the juice of the Siberian crab-apple (*Pyrus baccata*) can be highly recommended for the purpose, it being especially suitable for improving the keeping quality of fruit-wine. The juice of rhubarb stems may be added to that of elderberries, while the juice of gooseberries is suitable for mixing with that of mulberries.



Moreover, a combination of several juices may also be used; an excellent wine being, for instance, prepared from equal parts of blackberry, raspberry, currant, and strawberry-juice, with an addition of walnuts as given above. In the receipts for the different varieties given below, the customary addition of sugar for unmixed fermentation and the omission of salicylic acid is retained, but it may be repeated that with the assistance of these means the cost may be reduced one-half. In order to avoid repetition, the following general rules are here given, which hold good not only for the preparation of wine from small fruits, but also from stone-fruits.

The fruit to be used should be sound and ripe, though not over-ripe, and must be freed from adhering dirt by washing in warm water. Large quantities are best expressed by means of a press, while for small quantities a bag of coarse linen is sufficient, which is kneaded and squeezed until no more juice runs out. Over the residue pour as much hot water as juice is obtained, and after allowing it to stand for two hours press again and mix the juice obtained with the first. Now add sugar in the proportion of one pound to a quart of juice, and bring the whole into a thoroughly cleansed barrel previously rinsed out with salicylated water. Fermentation should take place in a room having a uniform temperature of from 59° to 64° F. During this process lay a piece of gauze upon the open bung-hole and secure it by means of a stone, piece of iron, etc., which prevents the access of foreign substances to the must. Every other day the barrel is filled up to the bung-hole with sugar-water prepared in the proportion of  $\frac{1}{2}$  lb. of sugar to 1 quart of water. As soon as the "hissing" in the barrel ceases, bung the barrel tightly and after 14 days draw off the contents into another barrel placed in the same room. After 6 months the wine can be drawn off into bottles, being, however, 8 days previously clarified with the whites of a dozen eggs or 1 oz. of isinglass slowly dissolved over a moderate fire in 1 pint of wine. Whatever fining is used, add it to the wine with constant stirring. If salicylic acid is to be used, it is best done in the man-

ner described for cider when the wine has acquired the desired degree of ripeness. The bottles should be rinsed with salicylated water and closed with corks previously soaked for a few hours in hot salicylated water. Sealing the bottles is not necessary, but in order to be sure that the corks fit closely, shake each bottle, with the neck downwards, with the right hand holding the left under the cork. If the slightest moisture is observed, the bottles must be recorked, as carelessness in this respect may cause a portion of the supply of wine to spoil. The corked bottles are laid in the cellar.

This general method, according to which all kinds of wine from small fruits can be prepared, may be supplemented by the following receipts:

*Currant Wine.*—Among all varieties of berries the currant contains the largest quantity of free acid, about 2 per cent., and comparatively little sugar, about 6 per cent. The proportion between these two principal constituents is very unfavorable for the manufacture of wine. The currant juice fermented by itself would yield a product which does not deserve that name.

Free the thoroughly ripe currants from the stems and after crushing press out the juice. To the residue add twice or three times as much water as juice obtained and after again pressing add the juice obtained to the first. Now examine the juice as to its content of acid and if necessary dilute further with water. Then calculate the sugar in the manner previously given. Sugar and acid having been brought to the right proportion, the juice is allowed to ferment.

Currant wine is frequently prepared as a sweet liqueur-wine, the following directions being much used for the purpose: Juice 100 parts, water 200, sugar 100. According to an analysis by Fresenius, the wine thus prepared showed after two years the following composition:—

Alcohol . . . . .	10.01
Free acid . . . . .	0.79
Sugar . . . . .	11.94
Water. . . . .	77.26
	<hr/>
	100.00

According to another receipt,  $17\frac{1}{2}$  lbs. of thoroughly ripe currants freed from the stems are bruised in a wooden vessel with the addition of  $3\frac{1}{4}$  quarts of water. The paste thus obtained is gradually brought into a bag of coarse linen, which is laid upon an oblique board, and pressed out by means of a rolling-pin. The press-residues are returned to the wooden vessel and, after adding 7 quarts of water, thoroughly worked with a pestle, and then again pressed in the above manner. The juice thus obtained is brought into a barrel having a capacity of  $34\frac{1}{2}$  quarts, a solution of 12 lbs. of sugar in 14 quarts of water is then added, and finally sufficient water to fill up the barrel to within 3 inches of the bung. After covering the bung-hole with a piece of gauze, the whole is allowed to ferment in a room having a temperature of from  $59^{\circ}$  to  $64^{\circ}$  F. When the principal fermentation is over, the barrel is entirely filled with water and closed with a cotton bung. The wine is then allowed to further ferment for six months in a cellar having a temperature of from  $54^{\circ}$  to  $59^{\circ}$  F., when it is drawn off into another barrel or into bottles. By adding to the fermenting juice  $\frac{1}{8}$  lb. of comminuted raisin stems a product closely resembling Tokay-wine is obtained.

A very strong beverage is obtained by adding to the expressed juice of currants twice the quantity of water and stirring in 2 tablespoonfuls of yeast. Allow the juice to ferment for 2 days; then strain it through a hair-sieve and after adding 1 lb. of sugar for every quart, allow it to ferment. When fermentation is nearly finished, add French brandy in the proportion of 1 quart to 40 quarts of the juice, and bung up the barrel two days later. The wine is ripe in four months.

According to another receipt the currants separated from



the stems, are pressed and the juice mixed with an equal quantity of water. Then add to each gallon of liquid  $2\frac{1}{2}$  lbs. of sugar, 2 ozs. of cream of tartar, and 1 oz. of pulverized nutmegs, with 1 quart of alcohol. Allow the whole to ferment, then fine with isinglass, draw off and bottle.

Another method is to express all the juice possible, then take an equal amount of boiling water, and pour it on the expressed fruit. Let it stand for 2 hours, squeeze out as much as there is of juice and mix; then add 4 lbs. of brown sugar to each gallon of the mixture; let it stand for 3 or 4 weeks, until fairly worked, with the bung out, and when it is done working, bung it up, then place it in a cool cellar.

*Strawberry-wine.*—For the preparation of wine very fragrant strawberries should be selected. The aroma of the strawberry is so delicate that it readily undergoes a change and soon disappears entirely. Hence to secure it and transfer it into the juice the strawberry requires special treatment, whereby neither the content of acid nor that of sugar is taken into consideration. This treatment consists in mixing the sound, ripe berries, without previous crushing or bruising, with the same weight of pulverized sugar and allowing the mixture to stand in a glass or stoneware vessel in a cool place until all the sugar is dissolved to a clear syrup in which the shrunk and tasteless berries float. To separate the latter, strain the juice through a woollen cloth previously rinsed with some lemon-juice or tartaric acid, dilute with the same quantity of water, bring the acid to 0.5 per cent., and subject the whole to fermentation in the usual manner at a temperature of from  $50^{\circ}$  to  $59^{\circ}$  F.

Some allow the berries to ferment with the juice, but the wine obtained is somewhat harsh and not as delicate.

By finally adding to the finished wine from 4 to 5 per cent. of rock-candy, a liqueur-wine is obtained which, as regards aroma, cannot be surpassed, and is especially liked by ladies.

Excellent strawberry wine is also obtained according to the following directions: Press out 10 lbs. of different varieties of

small and large cultivated strawberries, which give about  $2\frac{1}{2}$  quarts of juice. Pour water over the residue and press again, so as to obtain about 3 quarts more of juice or a total of  $5\frac{1}{2}$  quarts. Next dissolve 4 pounds of rock candy in 5 quarts of cold water, bring the solution, together with the  $5\frac{1}{2}$  quarts of juice, into a small cask, and allow the whole to ferment in a cellar having a temperature of  $61^{\circ}$  F. In four weeks the wine is ready for drawing off into bottles. It is of a beautiful pale-yellow color and possesses an excellent bouquet, and if made sparkling furnishes an excellent beverage.

According to a receipt in the "Weinzeitung," 40 quarts of strawberries and 41 quarts of water, with an addition of 12 lbs. of sugar,  $3\frac{1}{2}$  ozs. of tartar, and a gallon of whiskey free from fusel oil are allowed to ferment and the resulting wine is treated in the usual manner.

Another method is to pour 1 quart of hot water upon 1 quart of crushed strawberries and pressing out after allowing the mass to stand for 2 days. Then add to every quart of juice 1 lb. of sugar, and to every 40 quarts of juice the grated peel and juice of 2 lemons and 2 oranges and 4 quarts of French brandy. Allow the whole to ferment, and treat the resulting wine in the usual manner.

*Gooseberry-wine.*—The proportion between sugar and acid is somewhat more favorable in the gooseberry than in the currant, but not sufficiently so as that the pure juice would yield a good wine by fermentation. Hence the juice must be converted into suitable must, as regards sugar and acid, in accordance with the rules previously given. The yellow varieties are preferable, they alone having a distinctly vinous taste; the wine obtained from the red and green varieties being somewhat insipid. The juice is obtained in the same manner as from currants, the berries being bruised, the juice allowed to run off and the residue washed several times with water, so that each volume of juice receives an addition of 1 volume of water, though as the mixed juice has to be tested as to its content of acid, the direction in regard to the addition of

water need not be accurately followed. The must may contain as much as 30 per cent., because the fermentation of gooseberry-must is generally carried on in the warmer season of the year, so that all or the greater portion of the sugar ferments and the wine, on account of the quantity of alcohol formed, will keep for an almost indefinite time. Gooseberry-wine made from must rich in sugar generally acquires by age an odor of Madeira-wine, which frequently deceives even connoisseurs.

Gooseberry-wine, like currant-wine being liked sweet, a larger quantity of sugar may be added to the must from the start, though for a quicker process of fermentation it is better to add the desired quantity of sugar to the fermented wine. If the must has been made quite sweet, so that a wine rich in alcohol is formed, no fear need be had of the wine fermenting anew on account of the addition of sugar.

There are a number of receipts for the preparation of gooseberry-wine, but when more closely examined the products prepared according to them will be found either more or less rich in alcohol, or to contain more or less free acid, and to be either sweet or not sweet, so that the proportion can evidently be changed in any manner desired. It is further evident that nothing is gained thereby as regards quality, because the type for all artificial wines is grape wine obtained in a good season. In such wines the proportions between alcohol and free acid are well known and within such narrow limits that they cannot be essentially exceeded on either side, and they alone can serve as a basis for the rational preparation of gooseberry wine as well as of all artificial wines. With the aroma or bouquet which is to be imparted to such wine it is, of course, different; but no special directions are required, as every one manages it according to his own taste or according to that of those who buy and drink the wine. Thus it is also with the addition of sugar; one likes a sweet wine, the other one less sweet, and the third one without any sugar. The principal aim is to prepare a wine which contains the necessary quantity of alcohol to insure its



keeping properly, and the power of resistance against decomposing influences, and from which the greater portion of the fermentable substances is removed by fermentation. In most cases the natural conditions are of great use in this respect, for in order to decrease the content of free acid it becomes necessary to dilute the fruit juices, whereby the quantity of fermentable substances is also relatively decreased, and sometimes even to such an extent that they do not suffice for the complete fermentation of the sugar. Such wine, if not wanting in alcohol, will keep for an almost indefinite time and may be exposed to the access of air and a high temperature without the appearance of the formation of acetic acid.

*Gooseberry Champagne.*—The taste of this beverage closely resembles that of genuine champagne. There are several modes of its production. In France a light wine which does not contain too many fermentable substances is used. Somewhat less than 2 per cent. of sugar, or about 15 grammes to a bottle of 800 cubic centimeters' capacity, is dissolved in the wine and the latter drawn off into strong champagne bottles, which are then hermetically corked and tied with twine. The wine is then allowed to ferment in a room having a temperature of from 77° to 99° F. When fermentation is finished, the bottles are brought into a cool cellar and placed first horizontally and then gradually bottom uppermost so that the yeast may collect on the cork and the wine become clear. When all the yeast is precipitated to the neck of the bottle, the sediment is carefully removed—*degorgie* as it is termed—by first raising the string securing the cork and then the latter, the bottle being held in a horizontal position. The cork being no longer held by the string is forced out together with the deposit of yeast, while the clear wine impregnated with carbonic acid remains behind. To prevent the unavoidable loss of wine, the cork, together with the yeast and wine forced out, is collected in an upright barrel with a large aperture, towards which the mouth of the bottle is held during the operation.

The wine thus impregnated with carbonic acid, however, is

not yet champagne ; it only becoming so after the addition of a solution of fine rock-candy in brandy with which the bottle is filled up. Each bottle after receiving the necessary quantity of the solution, or *liqueur* as it is termed, is at once closed with a cork which is secured with twine or wire. Removing the deposit of yeast is the most difficult portion of this operation, long experience being required before the workman possesses the necessary skill.

According to another method, which is also called the impregnating method, the sugar required for sweetening is dissolved in the wine, and after clarifying the solution by filtering through paper pulp in a bag, or, if necessary, with some isinglass, it is taken to the impregnating apparatus, one similar to that used for mineral water answering the purpose. The wine is then saturated under a pressure of  $4\frac{1}{2}$  to 5 atmospheres with the desired quantity of carbonic acid and at once drawn off into bottles, which are corked and wired as above.

The advantage of this last named method consists in the rapidity with which champagne can be made, 30 to 36 months being required for the first method before the champagne is ready for transportation.

The following method is the most simple of all, but does not yield as fine a product. Each bottle is finished by itself and no special apparatus is required. The wine is sweetened and clarified in the same manner as in the impregnating method and then drawn off into bottles. In case the wine is not rich enough in alcohol, the content of the latter may be increased by 10 per cent.

After having filled the bottles about 1.52 cubic inches less than generally, add first to each bottle 11 drachms of pure crystallized bicarbonate of potash and immediately afterwards 1 oz. of pure crystallized tartaric acid in pieces. Then close the bottle with the cork and secure the latter by tying or wiring it crosswise. The potash and acid are now brought to solution by gently swinging the bottle to and fro, the contents becoming at the same time turbid by the separation of bitar-

trate of potash. By placing the bottle bottom upwards, the separated tartar is collected as much as possible upon the lower surface of the cork, and after the wine is clear, removed in the same manner as described in the first method. It is not absolutely necessary to remove all the tartar, as it settles on the bottom and the champagne will pour out clear.

According to any of these methods all fruit wines can be converted into champagne or sparkling wines.

Semler gives the following directions for the preparation of gooseberry champagne. Pour 20 quarts of warm water over 20 quarts of crushed gooseberries and add 6 lbs. of sugar,  $4\frac{1}{2}$  lbs. of honey, 1 oz. of pulverized tartar,  $\frac{1}{2}$  oz. of dried lemon peel, and  $\frac{1}{2}$  oz. of dried orange peel. After standing for two days strain the mixture through a hair-sieve into a barrel and add 2 quarts of French brandy. When the "hissing" in the barrel ceases, clarify the wine and after a few days draw it off into bottles, securing the corks with wire. Before filling the bottles throw a piece of sugar and  $\frac{1}{2}$  drachm of bicarbonate of soda into each.

*Raspberry Wine.*—Raspberries have such an agreeable and refreshing taste and odor that, while they are not very sweet and the proportion of acid to sugar is not very favorable, they are great favorites. Their aroma passes into the wine and would be even too predominant if for the preparation of wine the juice had not to be strongly diluted with water in order to decrease the acid.

As in all other fruit, the quality of the raspberry depends on the weather, and when this is favorable during the time of the development and maturing of the fruit, the latter is sweet and palatable, but in cold and wet seasons, sour and harsh. No other fruit suffers as much from such conditions as the raspberry.

We have the wild and cultivated raspberry. The wild raspberry is smaller than the cultivated but possesses a stronger aroma, but unfortunately is too frequently infested with the larva of many insects to render it always palatable. The cul-



tivated raspberry is considerably larger, and is less attacked by worms, but possesses less aroma and is frequently even watery.

To obtain the juice for the preparation of wine the thoroughly ripe raspberries are crushed to a paste in a wooden tub by means of a wooden pestle. To separate the grains, the paste is forced through a fine wire sieve, which, in order to protect it from the acid is best provided with a coat of asphalt or shellac varnish. It is, however, no disadvantage to allow the grains to ferment with the pulp, some tannin being thereby introduced into the wine, which under certain circumstances may be even desirable.

The content of acid in the raspberry varying considerably in different years, a test of the juice in this respect becomes absolutely necessary in order to enable one to dilute it in the correct proportion with water. For this purpose press out a small quantity of the crushed raspberries and determine the acid in the manner previously given. The sugar contained in the raspberry need not be taken into consideration, since by dilution it is reduced to 1 per cent. and still less. The must is simply brought up to 25 per cent. of fruit-sugar and allowed to ferment in the usual manner. The treatment of the wine after fermentation is the same as for other fruit wines.

*Blackberry wine* is prepared in the same manner as raspberry wine. Of the numerous directions for its preparation we give the following: Gather the berries on a dry day, crush them with the hand into a kettle, and add just enough hot water to cover the mass. Then add a handful of bruised raisins and a handful of strawberry leaves, from the heart of the mother plant, or, still better, from the suckers, and allow the mass to stand for four days, when a crust of yeast will have formed on the surface. The mass is now pressed out and sugar in the proportion of 1 pound to every 4 quarts added. Fermentation is allowed to go on for two weeks, when the barrel is bunged up and the wine drawn off after six months. During fermentation, and especially in the beginning of it, care must be had to fill up the barrel.

To make from blackberries a beverage resembling port-wine the following method is recommended : Press out the juice and allow it to stand for 36 hours. While fermenting during this time remove all scum from the surface. Now add of water, one-fourth the quantity of juice, and 3 pounds of brown sugar to every 4 quarts of fluid and filter after 32 hours. Fermentation, which requires but a few days, being finished, bung up the barrel tightly and after six months draw off the wine. The latter improves by age.

*Mulberry Wine.*—Press the juice from the fruit, dilute with the same quantity of water, add 1 pound of sugar for every quart of liquid, and boil the whole  $\frac{1}{4}$  hour. Then add for every 100 quarts, 3 quarts of alcohol,  $6\frac{1}{2}$  ounces of tartar, 1 ounce of cassia, and  $\frac{1}{2}$  ounce of bruised bitter almonds, and allow the whole to ferment. The further treatment of the wine is the same as for other fruit-wines.

*Elderberry Wine.*—Boil equal quantities of berries and water one-half hour, pour the whole into a hair-sieve, press the pulpy portion of the berries gently through with the hand and remove the residue. Compound the strained juice with sugar in the proportion of  $\frac{3}{4}$  pound to 1 quart, and boil 20 minutes. As soon as cool bring it into a barrel to ferment. Fermentation being finished, paste stiff brown paper over the bung-hole, and after eight weeks draw off the wine in bottles.

Another method is to boil 50 quarts of water, 10 quarts of elderberries, 40 pounds of sugar, 5 ounces of pulverized ginger, and  $2\frac{1}{2}$  ounces of cloves for 1 hour, with constant skimming. Then bring the liquid together with 4 pounds of crushed raisins into a barrel and allow it to ferment. At the termination of the fermentation it will yield a wine similar to the Cyprus or Greek-wine.

*Juniperberry Wine.*—70 quarts of water, 35 pounds of crushed raisins, 10 quarts of juniperberries, 4 ounces of tartar, 1 quart of French brandy, and a handful of fresh marjoram leaves are brought into a barrel and the mixture is allowed to ferment for 12 hours.

*Rhubarb Wine.*—Add to every 5 pounds of the thinly-sliced stalks  $2\frac{1}{2}$  quarts of soft water and bring the whole into a clean wooden vessel. Cover the latter and stir the contents with a wooden stick three times daily for one week. Then pass the fluid through a wide-meshed sieve and add to every 3 quarts, 4 pounds of white sugar, the juice of 2 lemons, and the peel of 1 lemon rubbed upon sugar. Allow the mixture to ferment in a barrel, and after clarifying, draw the wine off into bottles in March.

The variety of rhubarb known as Victoria is best adapted for the preparation of wine, which can also be effected according to the following directions: Cut up the stalks and express the juice. To every gallon of juice add 1 gallon of soft water and 7 pounds of brown sugar. Bring the mixture into a barrel and allow it to ferment until clear, with the bung out, keeping the barrel filled with sweetened water as it works over; then bung the barrel tightly or draw the wine off into bottles. It makes an agreeable and healthful wine affording a good profit, as nearly 1800 gallons of wine may be obtained from each acre of well-cultivated plants. The stalks will furnish about three-fourths their weight in juice.

*Tomato Wine.*—Press out the juice from ripe tomatoes, add to each quart of it 1 pound of brown sugar, and allow the whole to ferment. After three months the wine can be drawn off into bottles.

*Parsnip Wine.*—Cut 12 pounds of parsnips into thin pieces, add 15 quarts of water and boil until soft. Then press out the juice and after straining through a hair-sieve sweeten with  $\frac{3}{4}$  pound of sugar per quart. After again boiling for  $\frac{3}{4}$  hour it is brought, when cold, into a barrel and a tablespoonful of yeast is added. Stir the juice daily for 10 days, then bung up the barrel tightly and after six months draw off the wine into bottles.

In the same manner wine may be prepared from carrots, clover heads, corn stalks, etc. It is, however, recommended to add to the juice some aromatic substance such as a handful



of marjoram, almonds, plum kernels, currants, walnuts, ginger, or still better a few quarts of black currant juice.

*b. From Stone Fruits. Cherry Wine.*—Stone sweet cherries and after crushing the pulp to a paste allow it to ferment in stoneware pots for 12 hours. Then press out the juice, which is returned to the pots and allowed to stand until yeast fungi rise to the surface. Now add 1 pound of sugar to every 3 quarts of must, bring the latter into a barrel and allow it to ferment 8 days. Then rack the wine into bottles and keep in a cool place. The preceding is the method followed in England where pure cherry wine is made. It may, however, be remarked that it is somewhat insipid. A mixture of the juice of cherries with that of the raspberry or currant can, however, be highly recommended, it yielding a beverage similar to port wine. It is an American receipt and much preferable to the English. Press the freshly gathered cherries, black or red, but selecting those with the softest pulp, without crushing the stones. To the juice obtained add one-eighth of its quantity each of raspberry and black currant juice, and sweeten with lump sugar in the proportion of 1 pound to  $2\frac{1}{2}$  quarts of juice. The whole is then brought into a barrel to ferment. When fermentation is finished close the barrel tight and allow it to rest for three months. Then clarify the wine and draw it off into bottles. It is fit to drink in six weeks.

*Morello Wine.*—Press 60 pounds of morellos so as to crush the stones, mix the juice obtained with 20 quarts of sherry wine and the same quantity of warm water, and bring the whole into a barrel to ferment. Suspend in the barrel a bag containing  $1\frac{1}{4}$  ounces each of cinnamon, powdered nutmeg and mace, allowing it to remain until drawing off the wine. The latter is very palatable in two months after fermentation is finished.

*Plum Wine.*—Not all varieties of plums are suitable for the preparation of wine, but the Reine Claude and Mirabelle can be highly recommended, the latter especially making as spicy and agreeable wine as any variety of fruit. With the almost

innumerable varieties of plums it is not possible to say which are suitable for the preparation of wine and which are not. It can only be determined by experiment, though right sweet varieties only should be chosen. In this country the small sweet variety known as the wheat plum, etc., is frequently used for the purpose. The process is as follows: Stone the plums, then bruise the pulp, and add to every 8 pounds of the latter 3 quarts of hot water. After 2 days press out the juice and add to every two quarts of it one pound of sugar. Now bring the juice into a barrel in a cool room and add the crushed kernels of  $\frac{4}{5}$  of the stones. Allow the whole to ferment completely. After 12 months the wine is clarified and drawn off into bottles, each of which receives a small piece of sugar, which improves the keeping qualities of the wine.

*Apricot and Peach Wines.*—Both these varieties of fruit are used when nearly ripe. Remove the stones and crush the pulp to a paste. For every 8 pounds of the latter add 1 quart of fresh soft water, and let the mass stand 24 hours. Then press out the juice, add for every 2 quarts of it 1 pound of sugar, and allow it to ferment. During fermentation it is recommended to throw a handful of the crushed stones into the barrel, which gives to the product a more spicy flavor.

*Sloe or Wild Plum Wine.*—This beverage is not to be despised if prepared in the manner given for plum wine. The sloes must, however, remain on the bushes until after the first frost, which sweetens them.

## PART III.

### CANNING AND EVAPORATING OF FRUIT. MANUFACTURE OF CATSUPS, FRUIT- BUTTERS, MARMALADES, JELLIES, PICKLES, AND MUSTARD. PRES- ERVATION OF MEAT, FISH, AND EGGS.

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#### CHAPTER XXXI.

##### PRESERVATION OF FRUIT.

THE use of hermetically closed tin cans for preserving fruit has become of great commercial importance. Before discussing it, the various ways which have proved more or less satisfactory for household purposes will be briefly mentioned. The following rules apply, however, to all methods:—

1. The fruit must be gathered in dry weather and when free from dew. It is to be kept as free from dust as possible.
2. Absolutely sound fruit, not over-ripe, should only be selected.
3. The fruit should be preserved immediately after gathering.
4. The utensils used must be kept scrupulously clean.
5. The preserving vessels should not be placed directly upon the fire.
6. A good quality of white sugar only should be used ; brown sugar injuring the taste and color of the fruit.
7. Copper or enameled pans alone should be used for boil-



ing, if the latter is not effected in glass. The spoons should be of wood or of bone.

8. The jars or cans should be thoroughly rinsed, best with salicylated water, and if corks are to be used they should be perfectly sound and scalded in hot water to which some salicylic acid has been added.

9. Small jars or cans are preferable to large ones, and they should be kept in a dark, cool, dry place.

Bottled fruits should always be sterilized for 10 minutes, from the time the boiling-point is reached, in the case of  $\frac{1}{4}$ -bottles, 12 minutes for  $\frac{1}{2}$ -bottles, and 15 minutes for full-sized bottles. Only in the case of halved apricots and peaches and similar fruits that lie closely together, should a few minutes extra be allowed. Fruits that change color when heated, for instance, white pears, peaches and gooseberries should be separated after sterilizing in order to accelerate cooling.

First may be mentioned the old French method, known as *au Baine-Marie*, which on account of its simplicity, is still much used. Berries require no preparation, but peaches, apricots and plums must be stoned and halved, and cherries and small plums stoned. Apples and pears are peeled and quartered and immediately thrown into boiling water for 4 minutes to bleach. They are then laid a few minutes upon a sieve to dry, and brought, like other fruit, by means of a spoon into wide-necked glass jars which are filled to within 2 inches of the edge. In placing the fruit in the jar press it well together. The empty space is then filled up with hot syrup composed of 2 parts of sugar and 1 part of water, and the jars, after heating them somewhat upon a stove, are placed in boiling water for 8 minutes for kernel fruit and for 10 minutes for stone-fruit or berries. The jars are then immediately corked and sealed.

According to another French method, the flesh of the fruit is preserved without boiling. Stone-fruits and berries only can be used. The fruit is pressed through a hair-sieve and

the pulp mixed with an equal weight of pulverized sugar. The mixture is then brought into glass bottles, which are corked and sealed. This fruit-pulp keeps, however, only through the winter, or if kept in a cold place or in a refrigerator.

The following method gives better satisfaction: The fruit, such as cherries, berries, plums, peaches, apricots, etc., is, without the addition of water, brought into wide-necked glass jars in such a manner that a layer of fruit alternates with a layer of sugar, the top layer being sugar. The jars are then tied up with salicylated parchment paper, placed in a water-bath, and the water kept boiling for 15 to 30 minutes, according to the variety of fruit, small fruit requiring less time than large, and berries only about 16 minutes. The jars are then stored in a cool, dark place. For closing jars with narrow mouths corks are preferable. They are soaked in hot salicylated water and sealed.

Fruit thus preserved retains its fresh, natural appearance and keeps for a considerable time. If appearance is, however, of secondary consideration, it is better to boil the fruit, as is done with kernel fruit, melons, and all large varieties. The preparation for this method varies according to the nature of the fruit. Apples and pears must be peeled, and, if not too large, only cored, otherwise they have to be halved or quartered. Melons are peeled and cut into strips. Quinces are steamed until soft, then peeled as clean as possible, quartered, and the cores removed. After this preparation the fruit is brought into the preserving kettle and as much water as is necessary for boiling added. Boiling should be done very slowly and continued until the fruit commences to get soft. It should not be boiled too soft, but only sufficiently so to enable it to absorb the sugar-liquor. When this is the case the fruit is taken from the fire and strained, and with the liquor a syrup of the following composition is prepared: For each pound of fruit take one pound of sugar and soak it in  $\frac{1}{2}$  pint of the liquor. It is then placed upon the fire and the resulting

syrup skimmed. When it boils the fruit is introduced and slowly boiled, or rather simmered, because it must not fall to pieces, for five to ten minutes, according to its softer or harder nature. The fruit while still warm is then brought into the jars, in which no vacuum must remain. Hence they must be filled up to the cork, or if bladder or parchment paper is used, for closing them up to the rim. In the latter case it is advisable to place upon the surface a close-fitting piece of paper, previously saturated with a concentrated solution of salicylic acid in rum. Currants, blackberries and grapes are sometimes preserved in their natural clusters. They are first washed in fresh water, then slowly boiled soft, and strained. With the liquor a syrup of the previously mentioned composition is prepared, which is boiled and skimmed and poured upon the fruit in the jars.

Fine table pears are sometimes preserved in the following manner: Eight large pears are placed in a syrup prepared from 6 ounces of sugar, 3 ounces each of cloves and allspice,  $\frac{1}{2}$  pint of water, and  $\frac{1}{4}$  pint of port wine or other sweet red wine. In this syrup they are boiled very slowly—as much as 3 hours—until soft, and, while still warm, are brought together with the syrup into jars, which are treated in the manner previously described. By taking equal parts of pears and of fine plums a very beautiful product is obtained.

The boiling down of fruit in large stoneware pots is frequently accompanied by mishaps, and is more and more superseded by other methods. It consists in dissolving  $\frac{1}{2}$  to  $\frac{3}{4}$  pound of sugar in water and boiling the resulting syrup together with the fruit until the whole forms a jelly-like mass. While still warm the pots, which must be full, are tied up with bladder. A piece of salicylated paper should be placed upon the surface of the fruit before tying up the pots.

*Preserving in Air-Tight Cans.*—This method, as previously mentioned, has become of great commercial importance. The number of factories, briefly termed canneries, has largely increased, and not a few of them employ 1,000 hands during the



fall. Of course these factories do not limit themselves to the canning of fruit, as otherwise they would have to cease operations during the winter months, but that branch of the business preponderates over all others. The search after other suitable material is constantly more extended, and the trade-list of a large English factory now contains 200 different articles; including all Southern fruits, a portion of which is, singularly enough, returned in this state to the tropics. The American trade-lists embrace, as a rule, three groups, viz :

1. Apples, pears, peaches, apricots, plums, strawberries, raspberries, blackberries, currants, cranberries, whortleberries, nectarines, grapes, cherries, quinces, cocoanuts, pineapples, marmalade, jelly, green walnuts.

2. Peas, beans, beans with pork, corn, tomatoes, asparagus, carrots, onions, pickles, cauliflower, horseradish, mushrooms, catchups, succotash, plum-pudding, sweet potatoes.

3. All kinds of poultry, venison, salmon, lobster, crawfish, oysters, crabs, beef, mutton, pork, eels, salt-water fish, ham, pig's feet, beef tongue, lamb's tongue, frog legs, mussels, etc.

All the varieties of fruit named in the first group being not equally well adapted for canning, the less suitable kinds are only used in small quantities. Plums and cherries are preferably stoned, as well as peaches and apricots. Heart-cherries, black raspberries and whortleberries are the best suitable varieties of fruit for canning, as they lose their agreeable taste by steaming. Strawberries also become somewhat insipid, but red raspberries are excellent provided they are canned as soon as possible after being gathered. Blackberries are not quite so good though if, brought into the can immediately when plucked, they furnish an agreeable dish. Currants have too many seeds, and are better used for jelly. Black currants are well suited for canning, and in this state are much used by bakers for tarts. Gooseberries canned before entirely ripe are very good. Among the smaller stone fruit the Mazard cherry has few superiors, as if carefully canned it retains its shape, color, and aroma as on the tree. Most plums

are suitable for canning, provided they are stoned. Among the kernel fruits the quince occupies the first rank, as it is the only variety of fruit which gains by steaming. Pears are very suitable for canning, and even the inferior qualities can be used for the purpose. Apples, however, must be carefully selected, and only sweet varieties with firm flesh should be used. The Siberian crabapple can be highly recommended for the purpose.

As a general rule fruit for canning should have a firm flesh and fine aroma, these conditions being found in all the varieties preferred by the packers in the United States, whose canned goods can be found in every large city of the world.

Next to the variety of fruit, the cans are of the greatest importance. Much has been said and written in regard to them, and the discussion *pro* and *con* will very likely be continued. Glass jars have some advantages. They are comparatively cheap, allow of an inspection of their contents and the ready recognition of a leak, and are not attacked by the vegetable acid. But, nevertheless, they have not been introduced into general use because they are liable to break, and, being heavy, increase the cost of transportation, and, finally, it is difficult to close them air-tight. The sealing of a bottle with a narrow mouth is quite a different thing from sealing one with an aperture three inches in diameter. It may do for pickles, marmalade or jelly, but for preserved fruits which are to be transported long distances it cannot be depended on. The same objections may be made to stoneware jars, which possess the further disadvantage that their contents cannot be inspected and a leak is difficult to discover. Nevertheless, they are used by some large English factories for the reason, it is claimed, of keeping their products free from influences deleterious to health. To facilitate sealing, the jars are generally small—of about one pound capacity. Tin cans have many defects, but their use is very extensive, and in the United States they are almost exclusively employed. Complaint has been frequently made that the use of tin cans is deleterious to health because the tin contains lead,

which is dissolved by the vegetable acid and transferred to the fruit-syrup. In reply it has been said that only the inferior qualities of tin contain lead, and that only in an infinitesimal quantity ; but it cannot be denied that the solder may readily become injurious to health and in cases of poison investigated in the United States and England it could in every case be shown that the respective cans were soldered on the inside. The time is very likely not very distant when such soldering will be entirely done away with. To completely overcome all complaints against solder, as well as against a content of lead in the tin, cans are manufactured which are provided inside with a thin coating whereby the contents are protected from contact with the metal. The insoluble constituent of this coating consists of silicate of lime or glass-powder previously treated with hydrofluoric acid, while the soluble constituent is silicate of soda or of potash. Any silicate of earthy bases or metals may be used, or a precipitated gelatinous silicate. The alkali is fixed or removed by means of a bath containing a dilute solution of hydrofluosilicic acid, or a dilute solution with any other suitable acid. For preparing the composition mix the soluble with the insoluble silicate. The tin plates are coated with this mixture by means of a brush, or dipped in a bath of it and then dried by heat. The plates thus acquire a glass-like coating, which remains fixed no matter how the plates may be handled and worked.\*

In the canneries in the United States the cans are manufactured in a special department, and the division of labor is carried so far that every can passes through eight hands before it is finished ; and only with such a system is it possible to turn out large quantities in an incredibly short time. This far-reaching division of labor is, however, not limited to this department alone, but is the supreme law in the entire establishment. In the same department the solder is cut by a

\* In this country some packers of lobsters, shrimps, etc., line the cans with parchment paper.



machine into small three-cornered pieces. Each workman receives a certain quantity by weight of solder and of charcoal, with which he is expected to solder a certain number of cans. The workmen are paid by the piece, and each solderer has a number which is stamped in every can he solders, so that those which prove leaky may be returned to him for repair. By this system there is no waste of material, and the leaky cans do not exceed 5 in 1,000.

In another department the fruit is carefully inspected on long tables; the unsound being thrown out, and the sound turned over to the peelers and stoners, who of course work with the most improved machines. There are carriers bringing uninterruptedly fresh fruit, and off-bearers removing and sorting the waste. Nothing is thrown away, the waste being used partially in the manufacture of jelly and partially in distilling, and even the stones are utilized, as they are sold either to nurserymen or to chemical factories. Other workmen are occupied in placing the peeled and stoned fruit in the cans, which are handed over to boys, who place them upon small trucks running upon rails and transport them to the department where the filling in takes place. In the same department the syrup of sugar and water is prepared, but if the proportion of composition were asked a different answer would be received in every cannery. In regard to this point every manufacturer has his own ideas, which also extend to modifications for the different varieties of fruit. All manufacturers agree, however, that the best quality of white sugar should be used for light-colored fruits, and light-brown sugar for dark-colored, and that the syrup must be perfectly clear, and hence very carefully skimmed in boiling. In most factories the syrup used consists of 1 lb. of sugar dissolved in 1 pint of water. The filling of the cans with the fruit and syrup, the latter being generally kept warm, is effected with the assistance of scales, so that each can has exactly the weight upon which the selling price is based. The caps, previously provided with a hole the size of a small pea, are then soldered

upon the cans. The hole in the cap serves for the escape of the air during the succeeding process.

Different kinds of apparatus are used for the expulsion of the air by heating the cans. In large factories a steam retort is used which resembles in shape a ship's steam boiler. It is provided with a door closing air-tight, and is divided in the center so that it can be filled either half or entirely with steam, as may be required. The cans to the number of from 400 to 600 are placed upon trucks which run upon rails leading into the retort. Eight such trucks can be introduced at one time, so that it is possible to steam from 30,000 to 40,000 cans per day. The retort being filled, the door is closed and the pipe communicating with the steam boiler opened. The cans remain in the retort from 15 to 30 minutes, according to the variety of the fruit: Berries 15 minutes, stone-fruits 20, apples and pears 25, quinces and tomatoes 30. The door is then opened, and after the steam has somewhat dispersed the trucks are quickly pushed to the tin-shop, where the cap holes are soldered up. To cleanse the cans and make them shiny they are next put in a bath of soda water and then rinsed off with cold fresh water. They are then transferred to the store room, where they remain standing quietly for one week, when they are tested by striking the cap of each a short sharp blow with a wooden hammer. If everything is in order, the cap sinks slowly down, but if it is elastic and jumps back the can is what is called a "swellhead," and is returned to the tin-shop for repairs and is then again steamed. The perfect cans are labeled and packed and are now ready for market.

Another apparatus which can be highly recommended for small factories consists of a round iron plate resting upon a brick base about one foot high. Two round iron rods run up opposite to each other from the edge of this plate and serve as a support for a movable iron cylinder open at the bottom and closed on top. Upon the iron plate the cans are placed in the form of a pyramid, and the cylinder is then drawn down and screwed air-tight to the plate. A pipe communicating

with the steam-boiler enters the cylinder, and as soon as the latter is connected with the plate steam is admitted. After a certain time, which corresponds with that previously given, the steam is shut off, the cylinder pushed up, and the cans removed, the further treatment of which is the same as given above.

In some factories the cans are still heated, according to the old method, in boiling water. For this purpose the cans—100 at a time—are placed upon an iron plate attached to a steam-crane and submerged for 15 to 20 minutes in boiling water in a large shallow kettle. In this case the caps are not perforated, but soldered down air-tight. A workman watches the cans while they remain in the water and by means of a tool removes those from which small bubbles arise. Such cans being not air-tight are returned to the tin-shop for repairs.

The rest after being heated are also brought to the tin-shop, where the caps are perforated with a hole the size of a small pea, which is again soldered up after the escape of the heated air.

The canning of tomatoes, asparagus and other vegetables is effected in a similar manner except that no syrup is used. The Appert process for canning meat described later on under "Preservation of Meat, Fish and Eggs" is frequently used for the more expensive kinds of vegetables, such as asparagus, green peas, etc., glass vessels being generally used. The vegetables are first cleaned and trimmed, and are then covered with water in the vessels, with or without a little salt. Sticks of asparagus, or whole beans, are stood on end. The vessels are now lightly corked and boiled in a bath of concentrated brine, in which they are stood upright as fully immersed as possible. The bath is heated very slowly to avoid cracking the glass. It should take about two hours to bring the temperature to 212° F. The brine is then brought to the boil, whereupon the contents of the glasses will also boil. After they have been boiled for about ten minutes, the bath is allowed to cool to about 140° F., and the corks are driven in



tightly. Fused paraffin is then poured over them, and when the bath is quite cold, the glasses are taken out. The vegetables will then keep for as long as the vessels are unopened, for all ferments in them have been destroyed, and the paraffined cork prevents any more from getting access to them. The paraffin should come flush with the edge of the jar, and should be tied over with vegetable parchment to prevent it from cracking and flaking off. The top of the cork should be rough so that it may adhere better to the paraffin.

As the canning of tomatoes may serve as a type for all other vegetables a description of the process, for which we are indebted to Mr. Richard T. Starr, of Salem, N. J. is here given.

The tomato was for many years found only in hot-houses and conservatories of the rich. It was known as the love-apple and considered a curiosity. Our ancestors had no idea that this small red berry, for such was about its size, would ever, even under careful cultivation, become of mammoth size and form one of our most important articles of food. But such is actually the case to-day. The exact time when the now great industry of canning this vegetable commenced cannot be established with any certainty. The taste for the tomato seems to be an acquired one, and for years the industry struggled in its infancy until the breaking-out of the War of the Rebellion caused a demand that rapidly grew into gigantic proportions, and to-day finds the tomato-canning industry employing an army of men, women and children, while millions of dollars are invested in the payment of labor and the erection of plants.

In order that our readers may have a clear idea of the business we will commence with the beginning. Having made up his mind to engage in it on an average scale, the packer will first find a suitable plot of ground, on a navigable stream, if possible. Having secured this, the next thing is the erection of the buildings; these are generally one story in height and as large and roomy as the capital will warrant. The next step is to secure the requisite supply of fruit, and for

this purpose the farmers are drawn on and contracts entered into with them in which the packer agrees to take the entire marketable product of a certain number of acres, or else to take so many tons. These contracts are generally made about the first of the year, and as soon as the sun drives the frost from the ground the farmer prepares his beds and sows his seed. While the latter is growing, the land which is to be planted is heavily manured and plowed and carefully worked until it becomes mellow, and then hills about four feet apart are made, and into each one is put a small quantity of compost of phosphates. The tomato plants, having by this time grown to the height of 6 or 8 inches, are taken from the beds, and on a cloudy day, or the latter part of a bright day, transplanted and tended about as other growing crops. With a favorable season the farmer should commence delivering to the factory about the middle of August.

The arrangement of a canning factory is, of course, a matter of taste, but the most complete, in our opinion, is one where everything moves in a straight line, and in which none of the help are obliged to interfere with one another. The first thing to be done with a load of tomatoes is, of course, to weigh them, and for this purpose platform scales are built at an end door and the wagons driven on them. After being weighed the tomatoes are handed over to the scalding. Tomatoes arriving in all kinds of weather and conditions must, of course, not only be washed but scalded, so as to thoroughly loosen the skin from the pulp; and to do this quickly and properly, a heavy box of white pine is fitted with both steam and water pipes, and attached to it is an iron cradle swinging on hinges and raised and lowered by a wheel and pulley suspended above. On the back of this is placed a box, and as the farmer hands off his baskets they are emptied into this box, and at the command of the man at the rope, who is called the "scalding," they are dumped into the boiling water beneath. A few seconds suffice to clean and scald them; the cradle is then raised and the tomatoes are poured into kettles set in front of the scalding to receive them.

While this has been going on a group of women and girls have been filing into the factory and seating themselves along the trays that are to receive the tomatoes from the scalders. These trays are of different construction, but are similar as regards length, breadth and depth, the only difference being in the various ways of getting rid of the water and juice. This is generally done by making a slat frame fit in the bottom and over a trough fastened under the tray. This leads to a drain, which carries it to the creek or wherever else it is to go. At each tray are from ten to twelve women, each of them furnished by the packer with a bowl and knife, and provided at their own expense with a neat water-proof apron. The tomatoes are dumped from the kettles in front of them, and they remove rapidly the already loosened skins and cores and deposit the prepared fruit in a bucket sitting beside them. They become so efficient that a smart, active woman will frequently skin from 40 to 60 buckets a day, and as they receive 4 cents per bucket it will be seen they make fair wages. Standing just beyond the women are the machines which fill the cans. To describe them would be impossible, there being so many shapes and many makes. Some are very good, some very poor, every man thinks his the best, and so it goes; but in one respect they all agree: they have a hopper into which the fruit is poured from the buckets, and all have the plunger which forces the fruit into the cans; the treadles of some of them are moved by hand and some by steam. The machines rapidly fill can after can, which are then set on the "filling table" and receive "top them off," or in other words the fruit is cleared away from the top of the can so that the solder used in capping them will not become chilled. They are then placed in trays each holding either 10 or 12 cans and removed to the "wiping table," where everything is cleared from the top, wiped dry with sponges, and the cap placed over the opening. The "cappers" stand directly in front of the wiping table, and each one has his own fire-pot, irons, files, and everything he uses before him. Taking the tray, he rapidly applies by



means of a small brush the acid or flux necessary to make the solder flow freely around the cap, and then with the iron melts the solder and puts it in the groove. The can is then vented and is ready for the "bath." The baths, except in size, are constructed similarly to the scalding, and a thin cedar cover fits over each one. The cans are placed in wire or iron crates, lowered into the boiling water, and allowed to remain as long as necessary to cook them. The time of working varies in the different factories, but all the way from 30 to 50 minutes is required. They are then taken from the bath and placed on a slat-floor, where the air can pass through them, and when they are cold are "tested," generally by striking them with an awl. The testers become so expert that they can instantly detect by the sound an imperfect or leaking can; these are thrown out, mended, re-pressed, and put back in the pile. The cans are now ready for the next thing, which is labeling.

Labeling is done in different ways, and some canners with an idea of saving labor employ devices which are not only hard on the young girls who do the work, but which often result in much confusion and poor work. The best method is to divide the help into parties of five, one girl sitting on one side of the table with paste-pan, brush, and labels and the other four opposite her. The one girl, if quick and active, will paste the ends of the labels as fast as the other four can put them on the cans. The table is of course alongside the pile of cans, and two smart boys will place the cans on the table. As a girl labels a can she pushes it from her, when it is taken by the boxer, put in the box, and nailed up. This mode is simple and effective, and as the gang will label from 700 to 900 cases in a day the work progresses rapidly.

In many of the larger factories patent processing kettles, capping irons, and improved machinery are used, but as the result is, of course, the same, and they do not affect the mode of packing, it is not thought necessary to enter into any description of them.

In the foregoing an outline of the packing process has been

given, but nothing has been said of the many trials and vexations of a canner's life. If everything went always smoothly, it would be as pleasant as any other business, but it does not. The canner will early in the season employ his hands and commence in a small way. He may start and run only two or three hours, and for that length of time boilers will have to be fired up, help got together, and at the close the factory cleaned the same as if he had run the day out. Then, as the crop rapidly matures, work becomes heavier, and at last the inevitable "glut" commences, and he finds the products of 400 or 500 acres of perishable fruit at his doors, maybe 50 wagons, each owned by an impatient farmer standing in the street waiting his turn to unload. That is the time he has need of nerve; help must be secured, everything and everybody pushed to their utmost endurance, and from early morning until way into the night, day after day, the week goes on; help succumbs, and machinery breaks, but the factory must move in storm and in sunshine. The work must go on, and at last the agony is over, and the crop coming in again gradually gives a little relief to the overworked people.

It would be an impossibility to correctly state the amount of capital invested or the number of persons employed in the industry. The States of New Jersey, Maryland and Delaware pack a large proportion of the goods, the late falls and the nature of the soil being particularly well adapted for raising tomatoes.

In connection with the canning of tomatoes it may be of interest to our readers to give the preparation of

*Catchups.*—Under the name of catchup or catsup a thickly-fluid sauce comes into commerce, which is used as a condiment with meat, and the preparation of which has become of some importance. Everywhere where Anglo-Saxons reside catchup is found, though it has also been introduced on the continent of Europe and in the tropics. The varieties most liked are tomato and walnut catchups, and immense quantities of them are manufactured in the American canning establishments.

The mode of preparation is so simple that it can be introduced into every kitchen.

*Tomato Catchup.*—The receipts for making this favorite catchup are innumerable, and should those of every packer and housewife in the land be taken and put together they would make a good-sized volume.

In some factories where the tomatoes are peeled and either canned or made into some whole tomato product, such as chili sauce, the trimmings are made into catchup, all decayed portions being rejected. The trimmings are sometimes run to a chopper before going to the pulping machine. In some plants the stock is cooked before running it into the pulping machine, while in others the pulp is made from raw tomatoes. It makes little difference which method is used, so there is no material delay between the time of pulping and the using of the pulp. At some places the pulp is run as fast as made into a single vat and drawn out from the same during the day as needed. In this way the pulp is run with some that may have been in the vat for several hours, and there is a possibility of spoilage to begin with and consequently of some injury to the product. If the pulp is to be stored at all, a set of smaller vats is preferable, so that each vat as it is emptied can be cleaned out before a new lot is run in, thus checking any fermentation that might result due to the storing of the pulp in the same vat throughout the day's run.

The pulp obtained from the fruit, in making catchup is generally concentrated to about 50 or 25 per cent. of the original volume by boiling or the gravity method, the latter being employed by the majority of the plants making trimming pulp. At some plants it is customary to process the catchup after bottling while others find it unnecessary.

No receipt can be given that will suit all in regard to the amount of the different condiments to be used as each person has ideas of his own, but all catchup should be made hotter than desired, as it will undoubtedly lose some of its strength when it becomes cold. The best of spices and vinegar should



be used and every vessel into which it is put should be scrupulously clean and free from any mold or dust. Seal the bottles carefully, and if you have them thoroughly air-tight, the catchup will improve with age.

Below a few receipts for making catchup on a small scale are given.

I. Take 15 quarts of thoroughly ripe tomatoes, 4 tablespoonfuls each of black pepper, salt, and allspice, 8 red peppers, and 3 teaspoonfuls of mustard. The pepper and allspice must be ground fine and the whole boiled slowly 3 to 4 hours; then pass all through a fine sieve and when cold put it in bottles, which must be immediately sealed.

II. Boil 4 quarts of tomatoes together with 2 quarts of vinegar, 2 tablespoonfuls of red pepper, 4 tablespoonfuls of black pepper, 1 tablespoonful of cloves, 1 teaspoonful of salt, and 1 ground nutmeg, to a thick paste. Strain through a coarse-meshed sieve and sweeten the sauce obtained with  $\frac{1}{2}$  lb. of sugar. Fill in bottles and shake once every day for a week.

III. Cut up perfectly ripe tomatoes and place them upon the fire until they commence to bubble. Then take them from the fire, and when cool rub them with the hand through a hair-sieve and season according to the following proportions: For each quart of sauce add 1 teaspoonful of ground allspice, 1 teaspoonful of ground cloves, 1 tablespoonful of salt, and 1 quart of wine-vinegar. Stir the whole thoroughly together, replace it upon the fire, and boil for one hour, with constant stirring. When cool put the catchup in bottles and seal immediately.

*Walnut Catchup.*—I. In June, when the walnuts are still soft, take 10 dozen of them, and after crushing pour over them 2 quarts of wine-vinegar, add the following spices, all ground: 2 tablespoonfuls of black pepper,  $1\frac{1}{2}$  oz. of nutmeg, 40 cloves,  $\frac{1}{2}$  oz. of ginger,  $\frac{1}{4}$  oz. of mace, and boil the whole  $\frac{1}{2}$  hour, stirring constantly. When cold strain through a hair-sieve and put the catchup in bottles.

II. Crush about 10 dozen of young, soft walnuts, sprinkle

$\frac{3}{4}$  lb. of sugar over them, and then add 1 quart of vinegar. Let the whole stand six weeks, stirring frequently. Then strain through a bag, with constant pressing with the hand. Pour 1 pint of vinegar over the residue, let it stand over night, and strain again through the bag. Combine the fluid with that previously obtained and season with the following spices, all ground:  $1\frac{1}{2}$  oz. of black pepper,  $\frac{1}{2}$  oz. of nutmeg,  $\frac{1}{2}$  oz. of ginger,  $\frac{1}{4}$  oz. of mace, and 40 cloves. Then boil  $\frac{1}{2}$  hour, strain through a hair-sieve and bottle.

*Cucumber Catchup.*—Thoroughly ripe cucumbers, before turning yellow are peeled and grated upon a coarse grater. This paste is brought into a colander to allow the juice to run off, then pressed through a coarse hair-sieve to remove the seeds, and finally brought into small, wide-mouthed bottles, which are filled  $\frac{3}{4}$  full. The remaining space is filled up with good wine-vinegar. This catchup has the taste and odor of fresh cucumbers, and is used as a condiment with meat. Before bringing it to the table it is seasoned to taste with salt and pepper.

*Horseradish Catchup.*—The mode of preparation is the same as for the preceding, putting the grated mass in a colander and straining through a hair-sieve being, however, not necessary. Both varieties of catchup must be immediately corked, sealed, and kept in a cool place. Within the last few years both have been prepared on a large scale in the United States and England, and have become an article of export. They are packed in small, wide-mouthed bottles, sealed, and provided with gaily-colored labels. Some English factories use small earthenware pots of a cream color, closed with corks over which is tied strong colored paper. The pots are very good, but the manner of closing them is not; the corks should be sealed.

*Currant Catchup.*—Heat nearly to the boiling point, with constant stirring, 4 lbs. of thoroughly ripe currants together with  $1\frac{1}{2}$  lbs. of sugar. Then add 1 tablespoonful each of cinnamon, salt, cloves, and pepper—all finely pulverized—and 1 quart of vinegar. Boil the mixture one hour and then treat in the same manner as tomato catchup.

*Gooseberry Catchup.*—This product also comes into commerce under the name of “spiced gooseberries.” It is an excellent condiment with roast fowl. Take 6 quarts of gooseberries, ripe or unripe as may be desired, and carefully remove the stems and pistils. Then bring them into a kettle, and after pouring some water and scattering 5 lbs. of pulverized sugar over them, boil for  $1\frac{1}{2}$  hours. After boiling  $1\frac{1}{4}$  hours add 4 lbs. more of sugar and 1 tablespoonful each of allspice, cloves and cinnamon. The catchup is not strained, but brought at once and while warm into wide-mouthed bottles or pots, which are immediately corked and sealed. It is advisable before closing the bottles to lay a closely-fitting piece of salicylated paper upon the surface of the catchup. The bottles should be kept in a cool place.

It need scarcely be remarked that catchup can be prepared not only from the above, but from all varieties of fruit, and it is only necessary to take one of the above receipts as a type. But, with few exceptions, those given are the only catchups prepared on a large scale and brought into commerce.

Another subject which may be referred to in connection with the preservation of fruit is the preparation of

*Fruit-butter, Marmalade and Jelly*—*Fruit-butter.*—The manufacture of apple-butter, which may serve as a type of that of all other fruit-butters, is effected as follows: Fill the boiler two-thirds full with the juice of sweet and bitter-sweet apples in about the same proportion as given for the manufacture of cider. The other third of the boiler is filled up with slices of ripe, juicy apples, and the mixture boiled, with frequent stirring. When the slices of apples are so soft that they commence to fall to pieces, they are carefully removed from the boiler by means of a skimmer, care being had to allow the juice to run off. The same quantity of fresh slices of apples is then brought into the juice and boiled in the same manner as the preceding. When these have acquired the necessary degree of softness, the entire contents of the kettle, together with the slices of apples previously boiled, are brought into a



stoneware pot and allowed to stand covered for 12 hours. The mass is then replaced upon the fire and boiled, with constant stirring, until it has acquired the consistency of soft soap. If desired, it can at the same time be seasoned with cinnamon, nutmeg, etc. To prevent scorching, the second boiling is effected in vessels standing in boiling water.

In the same manner fruit-butter can be prepared from all varieties of fruit, pear or apple juice forming, however, always the boiling liquor. Apple and peach butters are commercially of the greatest importance, though butter of quinces, pears, blackberries, cherries, plums and cranberries is also manufactured on a large scale. Whortleberries, which grow in enormous quantities in some parts of the country, might also form an excellent material for this product. In the foregoing only the varieties are mentioned which are manufactured on a large scale by American and English factories that chiefly control the trade in fruit-butters, but these do not by any means exhaust the list. Green gages can, for instance, be highly recommended for the purpose.

The excellent product brought from France into commerce under the name of *raisiné* is prepared in the above manner by slowly boiling sliced apples and pears in unfermented grape-juice.

Fruit-butter is packed in wooden buckets of 5 or 10 lbs. capacity and in stoneware jars. Tin cans holding 2 lbs. are also sometimes used, but they are not liked. The buckets are slightly conical towards the top and are provided with a wire handle. Resinous wood should not be used in their construction, as it would impart an odor to the fruit-butter. The buckets are filled up to the edge, and a closely fitting round piece of paper previously saturated with concentrated solution of salicylic acid in whiskey is laid on top of the butter. The tight-fitting lid is placed upon the bucket without being sealed or otherwise closed. A large lable occupying the space between the lower and upper hoops finishes the packing.

*Marmalade.*—The same product is sometimes called mar-

malade and sometimes jam. The French prepare only marmalade, while the Englishman brings the same product into commerce as jam or as marmalade, just as it may suit him best, and the German is not much better. The term marmalade was originally applied to a jam prepared from quinces, it being derived from *marmelo*, the Portuguese word for quince. The term was gradually given to all jams in order to give them a more distinguished character, and this has led to a confusion of terms which sometimes extends even to jelly. There is, however, a wide distinction: Marmalade or jam is prepared from the pulp of fruit and jelly from the juice, while fruit-butter, as above indicated, is a blending of both with the omission of sugar.

For the manufacture of marmalade on a large scale all the rules and receipts can be condensed as follows: The fruit must be of excellent quality, entirely free from blemishes and washed perfectly clean. Kernel fruit is peeled, quartered and freed from the cores; peaches are also peeled, halved, and stoned; other stone-fruit is only stoned and halved, while berries are carefully freed from the stems. Melons and pumpkins are peeled and cut into small pieces. Rhubarb should not be washed but rubbed with a moist cloth and be then cut into small pieces. Tomatoes are to be peeled which is facilitated by previously placing them for one minute in hot water. Being thus prepared the fruit is brought into a copper kettle and as much water as is required for boiling added. While the fruit is boiling, weigh off as many pounds of white sugar as there is fruit, soak it in water, boil and skim carefully. The fruit should be boiled quickly, and when perfectly soft is allowed to cool off somewhat and then rubbed through a wide-meshed hair-sieve. The mass passing through the sieve is combined with the sugar and replaced upon the fire. The whole is then boiled with constant stirring, to the required consistency. The latter is tested by taking a small sample with a wooden or bone spoon—nothing else should be used—and if it draws threads between the fingers the boiler is removed

from the fire. The marmalade is then brought into straight jars, and after laying a piece of salicylated paper on top, the jars are tied up with white parchment paper or sometimes covered with a glass cover and labeled. It may be remarked that in the last stage of boiling the marmalade is sometimes flavored, which is generally effected by stirring in lemon juice, cinnamon, and nutmeg according to taste. The liquor obtained by boiling crushed kernels of plums or peaches is also often at the same time added as flavoring. Frequently the sugar is not treated as stated above, but added in the form of powder.

The quantity of sugar has above been given in the proportion of 1 lb. to 1 lb. of fruit. Though this is the customary rule, many manufacturers use only  $\frac{3}{4}$  lb. of sugar, a method which can be highly recommended. In fact there is frequently a perfect waste as regards the addition of sugar, some adding even  $1\frac{1}{2}$  lbs. of it to the pound, whereby the taste of fruit is entirely lost and the product, on account of its sweetness, to many becomes repugnant. It may be laid down as a rule that in all fruit boiling no more sugar than is absolutely necessary should be used. The secret of the great reputation the products of the principal American factories enjoy in all portions of the world is simply due to the fact that they use as little sugar as possible, whereby the products are rendered not only cheaper, but they retain their natural fruit taste, and that is what the consumer desires, and not a sugary paste having only the color of the preserved fruit. The durability of the product need not necessarily suffer if due care is exercised in its preparation. Marmalade should not be made, as it is only too frequently done, from fruit which has been gathered for several days and shows signs of decay. Fruit not over-ripe and freshly gathered should be used and the boiling finished as quickly as possible. By then rinsing the jars with salicylated water and covering the marmalade with a piece of paper saturated with concentrated solution of salicylic acid or with alcohol,  $\frac{3}{4}$  lb. of sugar to 1 lb. of fruit will be ample, and even  $\frac{1}{2}$  lb. with sweet



fruits such as pears, raspberries, etc. Independently of the saving of sugar, such marmalade will give better satisfaction than an article twice as sweet, and will keep well in a dark, cool place.

Some manufacturers use glucose in large quantities in making jams and marmalades. Some think it cheapens the bulk and causes it to congeal, while others claim that it causes the preserve to be heavy, syrupy and stringy.

In some factories *apple* pulp is used as foundation for cheap jams, the proportion of it employed varying according to what fruit is available. It is made by filling the steam-pan full of good cooking apples and, after turning on the steam, boiling them for 20 to 30 minutes. It is advisable to put some heavy weight on the cover of the steam-pan before turning on the steam to prevent it from being blown off. When boiling is finished take off the cover, and with a long paddle crush any apple that may have remained whole against the sides of the steam-pan. Then replace the cover and steam for about ten minutes more. The pulp is then ready for immediate use or storage.

All berry fruit-pulp will keep best when poured boiling hot into glass jars that have been rinsed out with boiling water. Fill the jars to the top and close at once. Sterilizing in tins alters the color of berry fruit-pulp. Lower grade fruit-pulp, of which large quantities are made for stock, may be stored in tins holding up to 50 lbs. as follows: The tins, which are soldered top and bottom, have a two-inch hole in the lid and are stood in a vessel of boiling water. When the fruit-pulp is boiling hot the tins are taken out in succession and filled up to the top, the lids being soldered on at once. The tins are then stood on their heads, so that the small amount of imprisoned air is compelled to rise through the boiling hot pulp, and is thus rendered innocuous. This method is perfectly reliable, and large quantities of pulp can thus be prepared for storage in a short time.

From France a very fine perfumed apple marmalade is

brought into commerce. It is prepared from equal parts of Calvilles and Pippins, and after boiling is sprinkled with rose-water or violet essence.

The term *tutti-frutti* is applied to marmalade prepared from a mixture of different kinds of fruit. As the name implies, it is of Italian origin. The composition is made according to taste and the fruits at disposal.

*English orange marmalade* is made from bitter oranges. Cut the fruit into halves without injuring the core, throw into boiling water, a few at a time, boil for several minutes and then cool them quickly. The flesh can be easily squeezed away from the rind. Heat the flesh to boiling with a sufficient quantity of water and pulp it in a mill, quarter the rind and cut it into thin slices, a special machine being used for this purpose. Blanch the slices until soft and lay them aside in a sieve. Next weigh out 30 parts of the pulp and 10 of the slices and mix thoroughly. In the meantime dissolve 54 parts of refined sugar and 6 of syrup and heat till they ball. Then add the pulp and rind and boil the whole to a finish. The marmalade should have a golden-yellow color and be perfectly clear. It is filled hot into the pots and fastened down when cold. The orange pulp is sometimes mixed with half its weight of apple pulp.

*Jelly.*—This product is, unfortunately, often made expensive and at the same time spoiled by too large an addition of sugar. Many housekeepers do not like to prepare jellies under the impression that they require too much sugar; but this is an error, because in France, in factories as well as in households, they use only  $\frac{1}{2}$  pound, or at the utmost  $\frac{3}{4}$  pound, of sugar to the pound of fruit, instead of 1 pound or even  $1\frac{1}{2}$  pounds, as is customary in England, Germany, and parts of the United States. Moreover, the apple-jelly which is made in the United States and sent to all parts of the world is made without any addition of sugar. Instead of apples, as the raw material, apple-juice is used, which must be perfectly sweet and treated immediately after it comes from the press. A moderate tem-

perature is absolutely necessary for success, for, if the juice commences to ferment—and it does very rapidly in warm weather—the keeping quality of the jelly is injured, except it be mixed with a considerable quantity of sugar. A temperature of  $41^{\circ}$  F. is considered the most suitable, and if it rises to above  $66^{\circ}$  F. the manufacture is at once stopped. The juice runs directly from the press into the boiler, under which a strong fire is kept because the starchy matters contained in the juice are only converted into sugar if the boiling down is quickly effected. For this reason shallow pans offering a large surface to the fire are used. When the juice commences to boil it is clarified, and the acid it contains neutralized by the addition of one teaspoonful of elutriated chalk to each quart of juice. The chalk weighed off in this proportion is mixed with the juice, and appears in a few minutes as a thick scum upon the surface, from which it is carefully removed with a skimmer. By this operation the jelly is clarified, and all the albuminous substances contained in it being removed by the chalk, filtering is not required. The process is similar to the defecation of the juice of sugar-cane and beets by lime. The juice is now boiled to the consistency of  $30^{\circ}$  or  $32^{\circ}$  B., which is found on cooling to be the proper point for perfect jelly. It is then filled direct from the pan into tumblers, which are treated in the same manner as marmalade jars.

Successful jelly boiling on a large scale is impossible without the use of the saccharometer. It is the only reliable guide for the addition of sugar, for if the product is to be protected from spoiling it must show from  $30^{\circ}$  to  $32^{\circ}$ . If this result can be reached without the addition of sugar, it is so much the better.

Pear and mulberry jellies are prepared in exactly the same manner as above. Other fruits containing more acid require an addition of sugar, especially currants, which next to apples and pears are most used for jelly, but in no case is the same weight of juice and sugar required.

To prepare jelly from berries and other small fruits, pour hot



water over the fruit in order to free it from adhering dirt and to facilitate the separation of the juice. When the water is cool take the berries out, express the juice, and bring the latter immediately into a copper or brass kettle over a lively fire. Then stir in pulverized sugar, the quantity of which varies according to the variety of fruit. For raspberries, strawberries, and blackberries  $\frac{1}{2}$  pound of sugar to the pound of juice will be sufficient, and  $\frac{2}{3}$  pound or at the utmost  $\frac{3}{4}$  pound for currants, barberries, elderberries and whortleberries. The sugar being added, stir in the chalk in the proportion previously given, and after allowing the juice to boil not longer than 15 minutes, take it from the fire and strain it at once into the glasses. In this manner a clear, beautiful jelly of an agreeable taste will be obtained. If, on the other hand, the juice is boiled slowly over a weak fire, the result will be a turbid product which has lost its fruity taste.

Stone-fruit is boiled, and after boiling it with a small quantity of water until soft, the juice is pressed out and  $\frac{2}{3}$  pound of sugar added for every pound. It should be boiled quickly, and not, as some receipts have it, for  $\frac{3}{4}$  hour. Quinces are peeled and then treated like stone-fruit. Rhubarb is cut into small pieces and then treated in the same manner. A quite good jelly can also be prepared from the medlar, provided it is allowed to become completely ripe, and is then slowly steamed with a very small quantity of water. When thoroughly soft the juice is pressed out and  $\frac{3}{4}$  pound of sugar added to each quart. The mass is sharply boiled for 20 minutes, when the result will be a clear jelly.

In France, as previously mentioned, perfumed marmalade is prepared from equal parts of Calvilles and Pippins. From the same material, which is considered best for the purpose, a perfumed jelly is also prepared. The apples are not peeled, but cut into slices, and boiled with a small quantity of water until soft enough to be pressed in a filter-bag. To every pound of juice  $\frac{1}{2}$  pound of sugar is added, and five minutes before the saccharometer indicates  $30^{\circ}$  B.,  $\frac{1}{4}$  or  $\frac{1}{2}$  pound of violet blos-

soms is stirred into the juice, a few drops of cochineal being generally added to improve the color. The jelly, when finished, is strained through a hair-sieve into wide-mouthed bottles, which are corked and sealed.

A jelly is made from raspberries, and sometimes also from strawberries and blackberries, in which the berries remain intact. The process consists in dissolving 2 pounds of white sugar in water and boiling until thickly fluid. Two pounds of berries are then brought into the kettle and carefully mixed with the sugar so as to avoid crushing. The kettle is then taken from the fire and allowed to stand covered for 15 minutes, when it is replaced on the fire and the sugar boiled up once more. The product is kept in jars well corked and sealed.

A description of the process of manufacturing apple jelly in one of the largest plants for that purpose may here be given.

The factory is located on a creek which affords the necessary power. A portion of the main floor, first story, is occupied as a saw-mill, the slabs furnishing fuel for the boiler furnace connected with the evaporating department. Just above the mill, along the bank of the pond and with one end projecting over the water, are arranged eight large bins holding from 500 to 1000 bushels each, into which the apples are delivered from the teams. The floor in each of these bins has a sharp pitch or inclination towards the water, and at the lower end is a gate through which the fruit is discharged, when wanted, into a large trough half submerged in the pond.

Upon hoisting a gate in the lower end of this trough considerable current is caused, and the water carries the fruit a distance of from 30 to 100 feet, and passes into the basement of the mill, where, tumbling down a four-foot perpendicular fall into a tank, tight in its lower half and slatted, so as to permit the escape of water and impurities, while in the upper half, the apples are thoroughly cleansed from all earthy or extraneous matter. Such is the friction caused by the concus-

sion of the fall, the rolling and rubbing of the apples together, and the pouring of the water, that decayed sections of the fruit are ground off and the rotten pulp passes away with other impurities. From this tank the apples are hoisted upon an endless chain elevator, with buckets in the form of a rake-head with iron teeth, permitting drainage and escape of water, to an upper story of the mill, whence by gravity they descend to the grater. The press is wholly of iron; all its motion, even to the turning of the screws, being actuated by the water-power.

The cheese is built up with layers inclosed in strong cotton cloth, which displaces the straw used in olden times and serves also to strain the juice. As it is expressed from the press tank the juice passes to a storage tank and thence to the defecator. This defecator is a copper pan 11 feet long and about 3 feet wide. At each end of this pan is placed a copper tube 3 feet inches in diameter and closed at both ends. Lying between and connecting these two are twelve tubes also of copper,  $1\frac{1}{2}$  inch in diameter, penetrating the larger tubes at equal distances from their upper and under surfaces, the smaller being parallel with each other and  $1\frac{1}{2}$  inch apart. When placed in position the larger tubes, which act as manifolds, supplying the smaller with steam, rest upon the bottom of the pan, and thus the smaller pipes have a space of  $\frac{3}{4}$  inch underneath their outer surfaces.

The apple-juice comes from the storage tank in a continuous stream about  $\frac{3}{8}$  inch in diameter. Steam is introduced to the large or manifold tubes, and from them distributed through the smaller ones at a pressure of from 25 to 30 lbs. per inch. Trap-valves are provided for the escape of water formed by condensation within the pipes.

The primary object of the defecator is to remove all impurities and perfectly clarify the liquid passing through it.

All portions of pomace and other minute particles of foreign matter, when heated, expand and float in the form of scum upon the surface of the juice. An ingeniously contrived float-



ing rake drags off this scum and delivers it over the side of the pan. To facilitate this removal, one side of the pan, commencing at a point just below the surface of the juice, is curved gently outward and upward, terminating in a slightly inclined plane, over the edge of which the scum is pushed by the rake into a trough and carried away.

A secondary purpose served by the defecator is that of reducing the juice by evaporation to a partial syrup of the specific gravity of about  $20^{\circ}$  B. When of this consistency the liquid is drawn from the bottom and the less agitated portion of the defecator by a syphon and thence carried to the evaporator, which is located upon the same framework and just below the defecator.

The evaporator consists of a separate system of six copper tubes, each 12 feet long and 3 inches in diameter. These are jacketed, or inclosed in an iron pipe of 4 inches internal diameter, fitted with steam-tight collars so as to leave half an inch space surrounding the copper tubes. The latter are open at both ends, permitting the admission and egress of the syrup and the escape of the steam caused by evaporation therefrom, and are arranged upon the frame so as to have a very slight inclination downward in the direction of the current, and each nearly underneath its predecessor in regular succession. Each is connected by an iron supply-pipe, having a steam-gauge or indicator attached, with a large manifold, and that by other pipes with a steam boiler of 30 horse-power capacity.

Steam being let on at from 25 to 30 lbs. pressure, the stream of syrup is received from the defecator through a strainer, which removes any impurity possibly remaining, into the upper evaporator tube; passing in a gentle flow through that, it is delivered into a funnel connected with the next tube below, and so back and forth through the whole system. The syrup enters the evaporator at a consistency of from  $20^{\circ}$  to  $23^{\circ}$  B., and emerges from the last tube, some three minutes later, at a consistency of from  $30^{\circ}$  to  $32^{\circ}$  B., which is found on cooling to be the proper point for perfect jelly. This

point is found to vary one or two degrees, according to the fermentation consequent upon bruises in handling the fruit, decay of the same, or any little delay in expressing the juice from the cheese. The least fermentation occasions the necessity for a lower reduction. To guard against this, no cheese is allowed to stand over night, no pomace left in the grater or vat, no juice in the tank; and further to provide against fermentation, a large water tank is located upon the roof and filled by a force-pump, and by means of hose connected with this, each grater, press, vat, tank, pipe, trough, or other article of machinery used can be thoroughly washed and cleansed. Hot water instead of juice is sometimes sent through the defecator, evaporator, etc., until all are thoroughly scalded and purified.

If the saccharometer shows too great or too little reduction, the matter is easily regulated by varying the steam pressure in the evaporator by means of a valve in the supply pipe.

If boiled cider instead of jelly is wanted for making pies, sauces, etc., it is drawn off from one of the upper evaporator tubes, according to the consistency desired; or it can be procured at the end of the process by simply reducing the steam pressure.

As the jelly emerges from the evaporator it is transferred to a tub holding some 50 gallons, and by mixing a little therein any slight variations in reduction or in the sweetness or sourness of the fruit used are equalized. From this it is drawn through faucets, while hot, into the various packages in which it is shipped to market.

A favorite form of package for family use is a nicely turned little wooden bucket with cover and bail, of two sizes, holding 5 and 10 pounds respectively. The smaller packages are shipped in cases for convenience in handling.

Each bushel of fruit will produce from 4 to 5 pounds of jelly, fruit ripening late in the season being more productive than other varieties. Crab-apples produce the finest jelly, sour-crabbed natural fruit makes the best-looking article, and a

mixture of all varieties gives most satisfactory results as to flavor and general quality.

*Saving of the Apple Seeds.*—As the pomace is shoveled from the finished cheese it is again ground under a toothed cylinder, and thence drops into large troughs through a succession of which a considerable stream of water is flowing. Here it is occasionally agitated by raking from the lower to the upper end of the trough, as the current carries it downward, and the apple seeds becoming disengaged drop to the bottom into still water while the pulp floats away upon the stream. A succession of troughs serves to remove nearly all the seeds.

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## CHAPTER XXXII.

### EVAPORATION OF FRUIT.

EVAPORATION is one of the most important methods employed for preserving fruit for any length of time. The reason for this can be readily given: The process does not require great technical skill; it excels in cheapness because neither vessels, sugar nor other auxiliaries are required; the product possesses excellent keeping qualities, retains its natural flavor, and by many is considered healthier and more agreeable than fruit preserved by any other method. While much fruit is still dried in the sun, and large quantities of it are marketed, the superiority of evaporated fruit has caused a large demand for it, and aside from the consumption in this country, large amounts are shipped abroad.

The Alden patent for evaporating fruit was granted about 40 years ago. Like all other new inventions, some years were required before its merits became thoroughly understood, though at the Paris Exposition of 1878 the first prize was unanimously awarded to the fruit dried by that process. Since then it has spread from California, where it was first



introduced, throughout the entire country, and though many types of evaporators are now in use, they are all based upon the same principle. At first only kernel and stone-fruits were evaporated, but at present the list includes almost every known fruit and vegetable.

Before entering upon a description of the apparatus and its use, an explanation of the principle upon which it is based and the theory of evaporating fruit will be given.

The object to be attained is not only to make the fruit keep, but also to retain the properties for which it is valued. This can only be reached by withdrawing the content of water, and at the same time converting a portion of the starch into sugar in as short a time as possible without boiling the fruit. The latter would injure the taste of the fruit, and slow drying gives a flavor calling to mind decay. The more quickly the watery portions are removed from thoroughly ripe fruit, the richer and more durable its taste will be; and the more completely the oxygen of the air is excluded during this process, the more perfectly will it retain its color. Rapidity of the drying process sometimes increases the content of sugar by 25 per cent., and this increase is in an exact proportion to the quicker or slower evaporation of the content of water, always provided, however, the fruit does not suffer injury from the heat.

Any one who has boiled down the juice of the maple, sorghum, sugar-cane, or sugar beet knows that with slow evaporation sugar is not formed, the content of sugar being then converted into acid. Now, the change of substance must be constantly kept in view: Starch is converted into sugar (in this case very largely already in the plant), sugar into alcohol, and alcohol into acetic acid. This experience must also hold good in drying fruit. The chemical process by which the content of starch of the fruit, when brought into a high temperature, is converted into sugar, is similar to that during the ripening process on the tree, only it takes place more rapidly.

A few days of warm sunshine produce sufficient sugar in

gooseberries and grapes to change the sour unpalatable fruits to a refreshing article of food. A few hours in an evaporating apparatus, in which the proper degree of heat is maintained, can produce a still greater change, provided the fruit be not placed in it before it has reached perfection in a natural manner. It must be remembered that  $212^{\circ}$  F. is the boiling point, and that subsequent treatment, no matter how careful, cannot restore the taste lost in such a temperature. Of no less importance is another point: The surface of the fruit to be dried must be kept moist and soft, so that the internal moisture may find a way by which it can readily and quickly escape, and a strong hot current of air must uninterruptedly pass over the fruit to carry off the escaping moisture. Hence, cold air must under no circumstances have access to drying fruit, and above the latter an aperture must be provided for the escape of the air saturated with moisture.

The apprehension that fruit cannot be dried in a hot moist apparatus is refuted by the well-known scientific fact, that air of the temperature of the freezing point absorbs  $\frac{1}{160}$  part of its weight of moisture, and that its capacity for absorption doubles with every  $15^{\circ}$  C. ( $27^{\circ}$  F.) of higher temperature. Thus, if the temperature is  $59^{\circ}$  F. it absorbs  $\frac{1}{80}$  parts of its weight of water,  $81^{\circ}$  F.  $\frac{1}{40}$  part,  $113^{\circ}$  F.  $\frac{1}{20}$  part,  $140^{\circ}$  F.  $\frac{1}{10}$  part,  $167^{\circ}$  F.  $\frac{1}{5}$  part,  $194^{\circ}$  F.  $\frac{4}{5}$  part, and  $221^{\circ}$  F. its own weight which is nearly equal to one pound of water to every  $\frac{1}{8}$  cubic foot of air.

The fruit would evidently never become dry if the air loaded with such moisture remained stationary, but set it in motion with a velocity of 880 feet per minute, which is equal to 20 miles per hour, and the cause of the rapid drying, or, in other words, of the withdrawal of water, becomes apparent. Now if we figure to ourselves an apparatus of 225 cubic feet content, the air heated in it to  $212^{\circ}$  F. contains, according to the above statement, 60 pounds of water, 50 pounds of which have been withdrawn from the fruit, while the remaining 10 pounds were contained in the air prior to its entrance into the apparatus,

because its temperature is supposed to be  $62.5^{\circ}$  F. With sufficient circulation to empty the apparatus every 20 minutes 150 pounds of water will each hour be carried away from a quantity of fruit supposed to amount to 800 pounds. Hence, in 5 hours, the time generally required for apples, 750 pounds of moisture could be removed if present.

Moreover, reference to a drying apparatus is not required to prove that heat alone does not suffice for drying. Is it not the wind which dries up the puddles after a rain more quickly than the hottest rays of the sun? The sun alone would effect nothing else but envelop the moist earth in a dense mantle of vapor destructive to both men and animals. Thus in the drying apparatus also it is rather the current of air which dries than the heat, but, of course, both must work in conjunction. The rapidity of the process prevents decay, and causes the color and aroma of the fresh fruit to be retained. The greater advantage of this rapidity consists, however, in the conversion of a considerable quantity of starch into sugar, which in sweet fruits, such as peaches, is sometimes formed in such abundance as to appear in small congealed drops upon the surface.

From the preceding it will also be readily understood why drying in the sun or in the oven must yield unsatisfactory results. Even with favorable weather the process lasts about 14 days. During this long time a fermentation sets in which partially destroys the content of sugar, and essentially changes the color and taste in an unfavorable direction. Such fruit when boiled tastes as if it had been preserved after the appearance of decay. Besides, during this process, the fruit is frequently selected as a breeding place by insects, in consequence of which it soon spoils, and when shipped to a distance resembles on arrival at its place of destination a heap of maggots. Such cases are not rare, especially if the dried fruit is shipped to tropical countries.

Drying in the oven has the disadvantage that the dry heat immediately closes the pores of the fruit, thereby rendering the escape of the internal moisture very difficult. If the heat is



not very strong the fruit remains moist in the interior, which causes it to spoil, and with a strong heat the surface carbonizes more or less. A portion of the sweetness is lost by being converted into caramel, the appearance of the fruit suffers by the tough shriveling of the surface, and the taste is injured by carbonization.

All these disadvantages are avoided by the modern evaporating process, which may be called a preservation of the fruit in its own juice with the assistance of steam.

A chemical analysis of a parcel of Baldwin apples shows best the changes effected in the composition of fruit by drying in the oven and by evaporation, and how the results with these two methods compare with each other. The first column gives the composition of 500 parts of fresh Baldwin apples. The second column gives the composition of the same parcel of apples after being reduced to 100 parts (loss of 400 parts of water) by drying in the oven, and a third column the result of 100 parts of the same parcel reduced by evaporation.

	Fresh.	Dried in the oven.	Evapo- rated.
Water (free and fixed). . . . .	411.15	12.42	16.62
Cellulose. . . . .	9.60	10.54	10.22
Starch . . . . .	32.95	30.95	29.75
Protein. . . . .	0.75	0.80	0.76
Pectine . . . . .	12.35	11.35	10.88
Gum . . . . .	6.75	7.22	4.33
Fruit acids . . . . .	6.70	4.88	3.43
Mineral constituents . . . . .	0.85	0.87	0.78
Chlorophyl . . . . .	0.15	0.12	0.15
Dextrin . . . . .	—	2.10	—
Grape sugar . . . . .	18.75	18.75	23.08
Volatile oils, traces . . . . .	—	—	—
	<hr/> 500.00	<hr/> 100.00	<hr/> 100.00

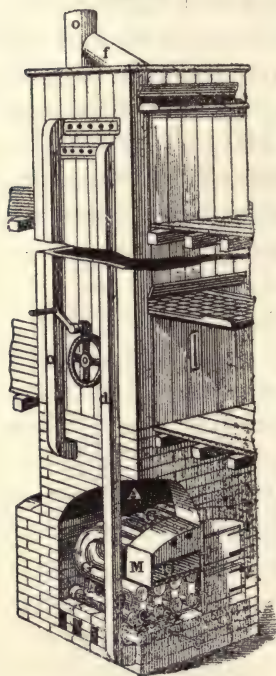
Attention must especially be drawn to the fact that dextrin, the formation of which is due to *dry* heat, is only found in the second column, and must be considered as an essential disadvantage of drying in the oven. The absence of this substance in evaporated fruit, as well as the presence of a larger

quantity of water (chemically fixed), is to be ascribed to the influence of moisture during evaporation.

As previously mentioned, many types of evaporators are now in use, some of them being small box-like structures of such a size that they can be placed on top of an ordinary cook-stove, while others have a sufficient capacity for handling fruit on a very large scale.

Fig. 99 shows an improved Alden evaporator which, like the Williams evaporator to be described later on, belongs to the type known as *tower evaporators*. *A* is the air-furnace

FIG. 99.



which is formed by the fire-box *D*, the ash-box *D*<sub>1</sub>, and the doubled horizontal pipes *G*, of which, according to the size of the apparatus, there are from 3 to 6, each 4 inches in diameter, and running parallel to each other. The products of combustion pass through them in the direction of the arrows, and escape through the smoke-pipe *O* at the back of the apparatus. The fire-box is surrounded by an air-space provided at *M* with apertures. Similar apertures to permit the entrance of cold air are provided on the side near the foot of the brick casing. The cold air comes first in contact with the lower, only moderately heated pipe, then rises to the second, and finally to the third and hottest series of pipes. It is thus gradually heated, and the pipes lying close together, each atom of air comes in contact with them,

which is considered a better mode of heating than by radiation, formerly used. The pipes are of cast-iron, and an escape of smoke into the drying-tower is impossible. By always keeping the pipes clean, which can be conveniently done, the

heat passes rapidly through their walls, and ascends immediately into the drying-tower without the possibility of super-heating.

The draught-pipe *d* connects the exit of the drying-tower with the fire-box of the furnace. The importance of this ventilation is sufficiently shown by the statement that for combustion 25,000 cubic feet of air per hour are required, which are introduced from the neighborhood of the opening of the tower through the pipe *d* into the fire-box. The removal of such a considerable quantity of air produces a vacuum in the upper portion of the tower, and consequently a very quick current of air over the trays of fruit in the tower—an absolute requirement for attaining great perfection in the art of drying fruit by evaporation. Besides, a saving of fuel is effected by the introduction of air, already heated, into the fire-box. The smoke-pipe *O* is surrounded by a wooden jacket, leaving a small intermediate space in which the heat radiating from the pipe collects, and is forced to enter the tower below the discharge-door. This also accelerates the current of air in the tower and prevents the condensation of the moisture, so that the fruit completely dries off in a short time. The branch-pipe *f* connects the opening of the tower with the smoke-pipe, which by its power of absorption also increases the current of air. The draught-pipe *c* is provided, as will be readily seen, for the purpose of uniformly distributing the heat in the tower.

The bulb of the thermometer, with which the apparatus is provided, is placed in the interior of the tower and the scale on the outside, so that the temperature can be read off without opening a door, whereby cold air would enter, which must be avoided. The air-furnace is constructed of brick, and the tower, as well as the draught-pipes *d* and *c* and the jacket of the smoke-pipe *O*, of double boards.

The hurdles or trays for the fruit consist of wooden frames with galvanized iron-wire bottoms. They hold from 20 to 60 lbs. of fruit each, and when charged are pushed through the door over the air-furnace into the tower, where they rest upon



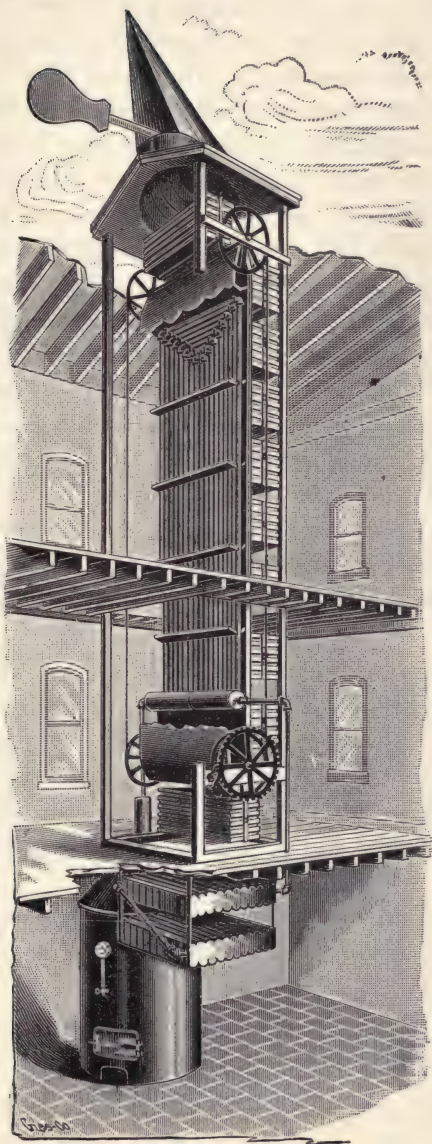
pins of an endless chain set in motion by a wheel, as seen in the illustration. The trays sit close to the walls on two sides of the tower, while in the other direction there is an interspace of two inches. The first tray is pushed tight against the back wall, the mentioned interspace thus remaining in front of the door.

After six to ten minutes, according to the variety of fruit, the tray is raised five inches by means of the endless chain; the second tray is then placed in position, but so that the above-mentioned intermediate space is at the back wall. At regular intervals the trays, when placed in position, are raised by the endless chain and the fresh trays pushed in, so that they touch alternately the front and back wall, the current of air being thus forced to ascend in a zigzag. When the tower is filled with trays it contains—taking apples as an example—from 1200 to 3000 lbs. of fruit. Every 50 lbs. of each yield from 40 to 45 lbs. of water, which ascends as vapor, which by surrounding the fruit with a moist mantle prevents its burning and keeps the pores open. When the tray first placed in position arrives at the discharge-door it has been in the tower for about five hours, and its contents have been converted into evaporated fruit which will keep for many years. Thus fruit can be gathered, evaporated and sold all in one day.

By considering the construction of the tower it will be seen that the fruit during its ascent remains in a uniform moisture and heat, so that up to the moment it is taken from the apparatus, its content of water can escape through the opened pores and, on the other hand, the heat can act to its very center. A uniform, perfect product can be obtained only by these means. When the fruit arrives at the discharge-door it is cool and as soft as fresh fruit.

Fig. 100 shows the Williams evaporator. It is heated by steam radiators located at the base of the vertical tower and has vertical radiating pipes up the center of the vertical tower, around which the trays of fruit revolve, with deflectors at intervals of two feet projecting from each side of said pipes to

FIG. 100.



direct the heat under the trays of fruit as they revolve around the pipes. (The trays and hanger are left out in the illustration to show the interior arrangement of the pipes.) These pipes or radiators extending up the tower from bottom to top produce a uniform heat the entire length of the tower, and increase the draught by increasing the heat at the top, which produces a more rapid circulation than when the heat is all at the bottom, as with the hot-air furnace; and the capacity of the apparatus is also increased in proportion to the increase of the heat and the draught through the tower. The trays of fruit in passing up the tower are exposed from one side to the pipes, and on descending are exposed from the other, which causes the fruit to dry uniformly. The tower being vertical the heat is utilized until it reaches the top. In this apparatus a very strong heat can be had throughout the entire length of the tower, without incurring any risk of fire from siftings from the trays, when drying cores and skins, falling on the hot-air furnace, which is always placed directly under the tower. Several sizes of this evaporator are manufactured.

The manner of operating the Alden apparatus is as follows: The maintenance of a uniform temperature in the tower being essential, the thermometer should indicate  $194^{\circ}$  to  $212^{\circ}$  F. Berries and stone fruit are to be kept somewhat cooler. The introduction of too much cold air into the air furnace must be avoided. As a rule an aperture two feet square suffices.

The upward motion of the trays must be effected at regular intervals. How long these intervals are to be, cannot be definitely stated, it depending on the content of water in the fruit and on the temperature of the tower. The following table may, however, serve as a guide:



Apples.....	interval 6 to 10 minutes.			
Pears .....	"	8	"	12
Peaches.....	"	12	"	20
Stoned plums.....	"	15	"	20
Apricots .....	"	8	"	15
Stoned cherries.....	"	10	"	20
Berries .....	"	10	"	20
Potatoes.....	"	6	"	8
Green corn .....	"	5	"	7
Onions.....	"	12	"	20
Tomatoes .....	"	20	"	25

It is supposed that the temperature directly above the air-furnace is  $212^{\circ}$  F., and it is best to keep it at that degree except for berries and stoned fruit, for which it may be from  $41^{\circ}$  to  $50^{\circ}$  less. As previously stated, it is an essential condition that the fruit should not boil. This will, however, not be the case at the temperature mentioned, because the fruit remains too short a time in it, and in rising upwards meets a somewhat more moderate heat. As a rule, it may be said that as high a temperature as possible is most advantageous, provided boiling be avoided.

The evaporated fruit, when taken from the tower, is spread out in an airy room, where it remains for a few hours to dry off previous to packing. Care must be had that during this time it does not come in contact with insects, and to prevent this the windows and air-holes should be provided with screens, or the fruit covered with mosquito netting. The fruit when ready for packing is put in boxes as follows: Line the box with colored paper with the ends projecting above the edge. Then fill the box with fruit. Kernel fruit is piled up about one inch above the edge of the box, while stone fruit is not piled so high, it being subsequently not subjected to pressure. To press down the contents even with the edge of the box a weight, or, still better, a press is used. After pressing, fold the ends of the paper over the fruit, nail down the lid, and put on the label:

Sliced evaporated apples are packed as follows: Line the

box with white paper, one piece on the bottom and four pieces on the sides long enough to fold over. Then nail down the lid, take off the bottom, and commence packing by placing one layer of slices in the manner of roof-tiles. Sufficient fruit to make up the required weight is then piled in, and after pressing down the box is nailed up and labeled. A general rule as regards weight has not been introduced, though in California all varieties of evaporated fruit are packed in boxes holding 50 pounds net.

In recent years tower evaporators have been largely superseded, especially for evaporating apples, by the *kiln evaporator*. This type is described by H. T. Gould\* as follows: "While the principles of construction of the different evaporators of this type are similar in all cases, the details of the arrangement of the appliances are endlessly varied.

"In constructing kilns the same general principles are followed, whether the evaporator is a small one with only a single kiln or an extensive establishment having several of them. The most satisfactory size of a kiln, all things considered, is about 20 feet square. This is a convenient size to fill, so far as the preparation of the fruit is concerned; the heat can be well regulated, made sufficiently intense for the purpose desired, and evenly distributed, so that the fruit will dry uniformly, and for various minor reasons a kiln of this size is a desirable 'unit' in the construction of evaporators of this type.

"A kiln consists essentially of a floor made of slats and placed over a furnace room or over a system of steam pipes. The floor is usually built from 10 to 12 feet above the floor of the furnace room. Provision should be made for regulating the heat by means of small openings in the base of the walls communicating with the outside which can be opened or closed as desired. The inflow of cold air can thus be regulated. Such control is specially desirable in windy weather.

\*U. S. Department of Agriculture. Farmer's Bulletin 291. Washington, 1907.

While many evaporators are constructed without special provision of this kind, it is an important point to have such openings, particularly if the walls are brick or otherwise made very tight, so that there is but little circulation of air.

"If the evaporator is a frame building, the walls of the furnace room may be well plastered or covered with asbestos paper to lessen the danger from fire, which may otherwise be great, because of the intense heat generated within them.

"If the walls, at least the portion below the kiln floor, are double, with an air-space between the two sides, the insulation will be more perfect than if they are solid or of only a single thickness, thus best conserving the heat and increasing the efficiency of the plant. The height of the walls of the kiln above the drying floor should be sufficient to permit an attendant to work on the floor conveniently and with comfort.

"Some means for the escape of the air laden with moisture from the fruit is necessary. This may be provided for by means of an opening in the roof, or a cupola-like ventilator may be built, the sides of which should consist of slats placed so that they overlap one another, as in an ordinary window-blind. Another form of ventilator is in the form of a tower about 3 feet square and extending 8 or 10 feet above the roof, which is sufficiently high to cause more or less draft, and hence augments the circulation of hot air through the fruit.

"The kiln floor is constructed of strips especially designed for the purpose. Such floors are generally made of poplar or basswood strips, seven-eighths of an inch thick, one inch wide on the top surface and one-half inch wide on the under side. In laying the floor these strips are placed one-eighth to one-fourth inch apart on the upper surface. This makes the space between them wider on the under side than on the upper, thus allowing the small particles of fruit which work down between them to drop through without clogging the intervening spaces.

"Satisfactory results are so dependent upon the heating apparatus that this becomes one of the most important features



of an evaporator. In the larger kiln evaporators, ordinary cast-iron stoves were formerly used considerably, two or more of them being frequently required to heat a single kiln, but these have largely gone out of use. In their stead large furnaces are now most commonly used. These are specially designed for the purpose and are provided with relatively large fire-pots, correspondingly large ash-pits, and large radiating surfaces. As it is necessary to burn a relatively large quantity of fuel in a given time, the size of the grate is made with this end in view. For a kiln floor 20 feet square, or 400 square feet of surface, the grate surface is usually about 3 feet in diameter, containing from 5 to 7 square feet.

“As to the most satisfactory length of pipe connecting the furnace and chimney, opinions differ. Perhaps the most common method of piping is as follows: The furnace, with two flanges for attaching the pipe, is placed in the center; the pipe from each flange is then extended to the side of the room opposite the chimney, and from this point the two sections, extending in opposite directions, follow the wall, at a distance of 2 or 3 feet from it, to the chimney. In a kiln 20 feet square, some 65 or 70 feet are thus required. Ten-inch pipe is a common size to use for this purpose. It is placed about 3 feet below the kiln floor.

“Some operators think that a better distribution of heat is obtained if the pipes extend back and forth, 2 or 3 feet apart, under the entire floor of the kiln, thus requiring 200 feet or more instead of the shorter length above suggested. The greater length, however, is less frequently used than the smaller.

“In some cases the heat is so intense directly over the furnace that the fruit dries more rapidly in the center of the floor than about the sides. To regulate this and make the drying as uniform as possible, a ‘deflector,’ consisting of a piece of sheet iron or tin several feet square, is attached to the floor directly above the furnace.

“Open grates, which in effect are furnaces with all parts

above the grates removed, are used occasionally and are recommended by some because they require less fuel, less attention to firing, and will dry the fruit in a shorter space of time. On the other hand so much dust rises from them that they are not used in making the best grade of fruit.

"In some respects a steam system is the most satisfactory method of heating, but it is comparatively little used, possibly due to the larger cost of installing such a system. In kiln evaporators the pipes are generally placed in as close proximity to the floor of the drying room as is convenient within a foot or even closer. That every steam pipe nearest the floor may supply the greatest amount of heat it should have its own return to the main return of the system. One inch pipe is generally used for such systems. No very definite data are available in regard to the amount necessary to supply the requisite heat. Several kilns, however, which are said to work admirably have about 600 running feet of pipe for every 100 square feet of floor space. One half of this is "riser," the other half "return".

"A convenient arrangement for an evaporator having four or five kilns is as follows: The kilns are built of brick and the apples are pared in an adjacent building. A bin built of slats for containing the apples in bulk extends the entire length of the building, except a small space in the center where a 5-horsepower gasoline engine is located, which furnishes power for running the parers, slicers and other machinery. The paring table is on the opposite side of the building, from which the fruit is taken by a carrier and elevated to a platform which is on the same level as the two bleachers between the evaporator and the paring shed. This carrier discharges the fruit into trays which are then placed by hand into one of the bleachers; from this they are taken to the slicer, located in a compartment just within the brick portion of the structure and with which all the kilns communicate, thus making it convenient after the fruit has been sliced.

"Other large establishments have the kilns arranged in a

series situated end to end. The fruit is pared on the first floor of an adjoining structure centrally located ; then elevated to the second floor which is on the same level as the kiln floors, where it is bleached and sliced. Communication is had with the kilns not adjacent to the floor on which the fruit is sliced, by means of a platform extending from this floor along the sides of the kilns and on the same level as the kiln floors."

For commercial purposes the selection of the varieties of fruit to be evaporated must be carefully made. This applies especially to apples and pears. As a rule, a product of high grade can be made from any sort which has a firm texture and bleaches to a satisfactory degree of whiteness. Many evaporating plants have, like the canning establishments, certain favorites, for instance, of apples, the Baldwin, Bell-flower, Pippin, Northern Spy, of pears, the Bartlett, Clapp's Favorite.

Apples are pared with a machine. So many different styles of apple parers for operating either by hand or power are in the market that it is difficult to say which is the best. The more recent patterns have two or even three forks for holding the apples while they are being pared. The attendant puts an apple on one of the forks while one on another fork is being peeled. The apples are cored in the same operation by an attachment applied to the paring machine for this purpose. The fruit is automatically forced from the fork and drops to the table where it is next taken in hand by the trimmers, who cut out with a straight-back sharp-pointed knife, worm-holes, decayed parts and other blemishes.

To make the fruit as white as possible it is usually bleached by subjecting it to the fumes of burning sulphur by means of a contrivance called a bleacher. The simplest form of construction consists of a box sufficiently long to meet the requirements, placed horizontally, and large enough in cross section to admit the boxes or crates in which the fruit is handled. Rollers are placed in the bottom, on which the crates rest,



which permit them to be moved along with but little friction. The crates are entered at one end of the bleacher, those previously put in being pushed along to make room for the following ones. The sulphur is usually burned immediately below the point where the fruit is put into the bleacher. A short piece of stovepipe is placed at the opposite end for the escape of the fumes after they have passed through the bleacher.

Another simple bleacher in which the fruit is handled in bulk (not in crates) consists essentially of a large square box, the interior of which is fitted with a series of inclined planes sloping in opposite directions to prevent the fruit from dropping to the bottom in a compact mass. The fruit is usually admitted at the top directly from the paring table. It then rolls from one inclined plane to another to the bottom, where there is the necessary opening, with means for closing it tightly to prevent the escape of the sulphur fumes, for removing the fruit when it is bleached. The sulphur is burned beneath the lowest inclined plane.

After bleaching, the fruit is sliced in a machine called a slicer, of which there are various styles. In general, a slicer consists of a table in which a series of knives is so arranged that when the apples are carried over them by a revolving arm they are cut into slices about  $\frac{1}{4}$  inch in thickness. In the kiln-evaporator the sliced fruit is evenly spread on the floor to the depth of from 4 to 6 inches. It is a common practice to treat the floor of the kilns occasionally with tallow to prevent the fruit from sticking to it. Sometimes a mixture of equal parts of tallow and boiled linseed oil is used for this purpose.

No definite rules can be given regarding the temperature to be maintained in the kiln, this being largely a matter of experience. Some operators consider  $150^{\circ}$  F. a suitable temperature when the fruit is first put into the drying compartment, dropping to about  $125^{\circ}$  F. as the drying process nears completion.

The fruit while drying in the kiln has to be occasionally

turned to prevent it from burning and from sticking to the floor. For the first five or six hours it is generally turned every two hours or so, and more frequently as it becomes drier, until perhaps it may require turning every half-hour when nearly dry.

When drying in the tower evaporator the trays or racks must not be too heavily loaded with fruit. Stone-fruit not freed from the stones is placed close together with the stem ends upwards, but only in one layer. Plums after evaporating are generally brought into a bath of sugar-water to give them a lustrous and uniformly dark appearance. For this purpose brown sugar is dissolved in an equal quantity of hot water, and the prunes in a wire basket are submerged in the bath for half an hour. They are then spread out upon hurdles and packed when perfectly dry. Quartered or halved stoned-fruit, as well as sliced apples, are placed close together, edge upward, until the bottom of the tray is covered. Sliced pears are arranged in a similar manner. Of berries, several layers an inch deep may be made, but they must be covered with tissue paper. Grapes are but seldom converted into raisins in the evaporating apparatus, because the process would require 40 hours, it being impossible to use a temperature exceeding 167° F. Hence it is considered more advantageous to dry grapes in the sun. The well-known Malaga raisins are obtained by allowing the bunches of grapes to dry in the air. They are dipped for an instant in boiling water to sterilize them and then dried on straw in the sun. When the grapes have shrunk to a third or half of their original volume, the best are packed in the original bunches, but the inferior raisins are picked from the stalks before packing. The richer the grapes are in sugar, the less drying they need. In Spain the bunches are dipped into a boiling lye of wood ashes on which a little oil is floating. They are dipped and removed as quickly as possible, and the trace of oil that adheres to them gives a characteristic luster.

Tomatoes to be evaporated in the tower evaporator are

peeled but not sliced, and placed close together in one layer in the trays. Pumpkins are peeled and cut in pieces two or three inches thick. For several years a flour has been made from the dried pieces, which serves as a substitute for rice flour. Sweet potatoes are treated in a similar manner, their flour serving as a substitute for chicory.

Green corn is first steamed on the ear for not more than five minutes. The grains are then picked off, placed in two-inch-deep layers in the trays and thoroughly evaporated, but not at too high a temperature, 185° to 194° F. being sufficient. When dry they are rubbed and passed through a fanning-mill to remove the hulls loosened by rubbing. The corn is packed in boxes holding 10, 20 and 50 lbs. each.

The following must also be steamed before evaporating: Green peas and beans, asparagus, beets, carrots, lettuce, cabbage and parsnips. Vegetables are cut up with a cabbage-cutter, and roots in slices like apples.

Onions are first freed from their external red or yellow peel and then cut into slices one-fourth inch thick with a cabbage-cutter. The slices are steamed for five minutes with a suitable steaming apparatus, which is best effected by spreading the slices in a two-inch-deep layer in the trays, placing the latter in the steaming apparatus, and immediately after the above-mentioned time in the evaporator. They are packed in tin boxes holding 50 lbs. each, which are placed in a wooden box. By evaporation, 100 lbs. of onions are reduced to 12 lbs.

Potatoes must be thoroughly washed. This is best effected in a cradle, the bottom of which is provided with wide perforations so that the water, constantly pouring in, can run off quickly. The potatoes are then placed in trays, and from four to six of the latter, according to the size of the steaming apparatus, brought into the boiler. Steam is then admitted, and after 35 minutes the potatoes are taken out, care being had, however, not to steam them too much, as otherwise they become of no value for the evaporating process. The loosened peels are then rubbed off with the hand, and the peeled pota-



atoes brought into a press, the bottom of which consists of a perforated wooden plate or of woven wires. The lid must fit tight into the interior walls of the press, so that the entire mass of potatoes falls coarsely crushed through the bottom. The crushed potatoes are placed in layers two or three inches deep in the trays and leveled with an instrument made by driving small nails into a board so that their points project one-half inch. They are then evaporated at not too high a temperature—185° F. is sufficient—to prevent scorching ; taking care, however, to dry them through. The evaporated mass is coarsely ground in a suitable mill, and the resulting flour packed in zinc canisters, holding 28 and 56 lbs. each. Two such canisters are placed in a wooden box and are then ready for shipment.

It is of the utmost importance to select only perfectly sound potatoes and remove all which sour or are injured in any other way during the process. Success depends on the rapidity and regularity from the commencement to the end of the process. All potatoes which become cold before being brought into the evaporating apparatus are worthless, and the same may be said of those which have been steamed too long ; they are converted into paste.

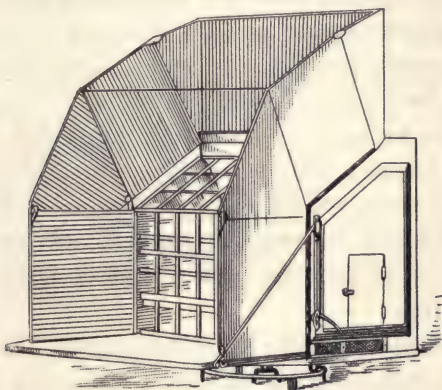
Attention may be drawn to a sun-drying apparatus shown in Fig. 101, which may be recommended to those who do not wish to employ artificial heat, and are forced to give the preference to as cheap an apparatus as possible. The apparatus is constructed of boards and window-glass. The board walls, which are somewhat inclined outwardly, project above the panes of glass and serve, as is readily seen, for catching the rays of the sun. They are lined inside with tin, thus becoming reflectors. The side door serves as an entrance to the apparatus when the panes of glass are to be cleansed or repairs are to be made in the interior. The trays containing the fruit are pushed in from the back, the entrance of each tray being covered by a wooden flap. According to the size of the apparatus two or three rows, each consisting of twelve trays, are

placed alongside each other. Above the uppermost entrances for the trays are slides, which can be opened or closed according to whether the heat in the interior is to be increased or moderated.

The apparatus stands upon a turn-table, so that the front can from morning to evening be exposed to the full rays of the sun. When the latter no longer reach the apparatus the reflectors, which are hinged, are laid over the panes of glass, which prevents the radiation of heat, and protects the fruit from dew.

The time required for drying fruit in this apparatus cannot

FIG. 101.



be definitely stated, but on an unclouded, hot summer day, apples pared by a machine can be dried in eight hours. The product obtained is not of as good a quality as evaporated fruit, but it is incomparably superior to that produced by the primitive method of drying in the open air or in the oven.

In conclusion it remains to say a few words about drying fruit in the oven, and we describe the French method, which is decidedly the best, as proved by the prunes brought into market from that country. The prunes having been sorted by a machine into three qualities are placed upon trays and exposed to the sun until the skin commences to shrivel. They

are then placed in a bake-oven previously used for baking bread. If no bread is to be baked, the oven is very moderately heated to prevent the rapid closing of the pores and the formation of a crust upon the surface. They are allowed to remain in the oven for 12 hours, when they are taken out, and when perfectly cold, moistened with alum water and replaced in the oven, which must now be somewhat hotter. After 12 hours they are again taken out, moistened with alum water, and replaced for the third and last time, together with a dish full of water, in the oven, which must now be still hotter than before. The prunes when taken from the oven are submerged for a short time in a bath of sugar-water, and are then packed in boxes. It will be seen that this process is quite tedious, and the product is not so good as that obtained by evaporation.

Besides prunes, the French bring into the market dried pears, which have also become celebrated. The process is as follows: Fine table-pears are pared, quartered, and boiled in sugar syrup for five minutes. They are then placed in a moderately warm oven, where they remain for 12 hours; they are then taken out, allowed to cool off, and replaced in the oven, which must now be hotter than the first time, until sufficiently dried.

The French method can be recommended, but it would be still better if it were executed in the improved manner practiced here and there in central England and in the New England States. This improvement consists in the previous boiling of the fruit, which must, however, not be continued longer than five minutes. The fruit is not gradually heated, but submerged in boiling water for five minutes, and, without being allowed to cool, brought at once into a moderately hot oven. Steaming instead of boiling the fruit is still better. It should be exposed to the steam for not longer than five minutes, and must then as quickly as possible be brought into a moderately hot oven.



## CHAPTER XXXIII.

## PREPARATION OF PICKLES AND MUSTARDS.

*Pickles.*—Enormous quantities of pickles are brought into commerce, especially by American and English factories. The most remarkable varieties are piccalilli or Indian pickles, mixed pickles and walnut pickles. The packing is always the same, some of the oldest and largest English factories still adhering to stoneware pots, which have the advantage of entirely excluding the light from the product, thus contributing to its keeping quality. Both the French and Americans use glass bottles, the chief difference being in the diameter of the mouth, which is smaller in the American bottles. The latter style is to be preferred, because in the former the pickles, when frequently opened, are more exposed to the air than is good for them. Stone pots, which are no doubt best for family use, are too expensive for commercial purposes, not only as regards the first cost, but also on account of their weight, which increases the cost of transportation. The bottles are always provided with neat labels, and the corks generally covered with tin-foil.

The following general rules apply to the preparation of pickles: The best quality of fruit must be gathered at the right time, washed in fresh cold well-water, and placed for some time in strong brine. They are then laid upon fruit hurdles to completely dry in the air, and finally brought into the bottles, which must be nearly filled. The interspaces are then filled up with hot-spiced vinegar, and the bottles immediately corked, and, when cold, sealed. Strong vinegar must be used, the manufacturers generally employing wine-vinegar, known in commerce as No. 24. Fruit-vinegar, clarified and spiced and evaporated to three-fourths its volume, also answers very well. Pickles for immediate use are soaked in hot brine, but as a commercial article they must be treated with cold brine only.

Moreover, hot brine must not be used for vegetables of a soft and juicy nature such as cabbage and cauliflower ; and besides, cold or only slightly heated vinegar should be poured over such articles. Soft and delicate fruits must, as a rule, not remain as long in the brine as hard and coarse-fibred ones ; and the softest are most advantageously pickled by pouring cold spiced vinegar over them. The same may be said of red beets and other roots which are cut into strips. Sometimes the spice is put whole into the bottle, but it is better and more economical to bring it powdered into the vinegar while heating the latter, or if the vinegar is to be used cold, to previously boil the powdered spice in a small portion of the vinegar, and when cold add it to the rest. The spiced vinegar is prepared as follows :—

To 1 quart of vinegar add  $2\frac{1}{2}$  ounces of salt,  $\frac{1}{2}$  ounce of black pepper, and  $2\frac{1}{2}$  ounces of ginger. Let the mixture boil up once or twice in an enameled iron pot, filter through a flannel cloth, and pour the liquid, hot or cold, over the fruit.

For a more strongly spiced vinegar reduce in a mortar 2 ounces of black pepper, 1 ounce of ginger, and  $\frac{1}{2}$  drachm of cayenne pepper, and for walnuts, 1 ounce of eschalots, and add to the mixture in a stoneware pot, 1 pint of vinegar, and tie up the pot with a bladder. Place the pot for three days near the fire, shaking it several times, and then pour the contents upon the fruits by allowing it to run through a filtering cloth.

In the preparation of pickles the use of metallic vessels must be avoided, the vinegar as well as the brine dissolving copper, brass, and zinc, and becoming thereby poisonous. Ordinary earthen pots should also be mistrusted. Stoneware pots, which can be heated in a water-bath or upon a stove, are best for the purpose. Moreover, air and light must be kept away from the pickles as much as possible, and they should be touched only with wooden or bone spoons. An essential condition for success is to treat the fruits immediately after being gathered. The method of some manufacturers, who add verdigris to the pickles or boil the vinegar in a copper boiler until it is suffi-

ciently "greenish" to communicate its color to the product, cannot be too strongly condemned. That this crime against the health of the consumer is unfortunately committed to a considerable extent is conclusively proved by numerous chemical examinations made in the large cities of Europe and the United States, and undertaken with the laudable purpose of bringing the adulterators of food to justice. Many of the pickles in the market and most of the imported canned peas contain copper, and this notwithstanding the fact that there are very innocent means for coloring pickles green, it being only necessary to put a handful of spinach or grape leaves in the boiling vinegar, which acquires thereby a green coloration and communicates it later on to the pickles.

The following list comprises the fruits and vegetables which are chiefly used for the preparation of pickles in factories:

*Barberries.*—The berries are gathered before they are ripe and washed with salt water. The vinegar is added cold.

*Beans.*—Cold vinegar is poured over the young pods, previously soaked in cold water.

*Cabbage, Red and White.*—The heads are cut up into fine strips, which are placed in a strong brine for two days, then dried upon hurdles for twelve hours, next brought into bottles, and after pouring cold vinegar upon them, at once sealed up.

*Cauliflower.*—The heads are broken up into small pieces, which are placed in brine, and finally treated with hot vinegar.

*Cucumbers.*—Young cucumbers are placed in salt water for one week. The brine is then poured off, and after being made boiling hot is poured back over the cucumbers. The next day the cucumbers are dried upon a sieve, slightly rubbed off with a cloth, and then boiling vinegar is poured over them.

*Elderberry Flowers.*—The umbels are gathered just before the flowers open, and treated in the same manner as cauliflower. These pickles are much liked in England.

*English Bamboo.*—Young elder shoots are freed from the bark, placed in a brine for 12 hours, and after drying brought



into bottles and hot vinegar is poured over them. They are highly esteemed as an addition to boiled mutton.

*Gooseberries.*—The unripe fruits are treated like cauliflower.

*Mixed pickles* are a mixture of young, tender vegetables, preserved separately, each at the proper season, and stored for mixing later on. Small gherkins, young corn cobs, pickling onions, green and red capsicums, cauliflower, young beans, French beans and French carrots form the chief ingredients of this mixture, large capers and salted olives being also used by some makers. All the vegetables are highly blanched and left to stand for several days in strong brine in order to ensure their keeping. They are then put in jars with good vinegar, warmed up in the water-bath and closed up. The vegetables may, however, be stored separately in brine in case of very large quantities.

For packing in bottles and other containers, good 4 per cent. wine-vinegar is used, this being spiced with an extract prepared as follows: To 5 gallons of vinegar add 36 ozs. of black pepper, 18 ozs. of ginger, 18 ozs. of salt, 10 ozs. of pimento, 6 ozs. of white pepper, a few laurel leaves and a little tarragon, allowing the mixture to stand for 14 days in a warm place and then filtering. The filled bottles should be carefully heated on the water-bath before they are closed.

*Picalilli.*—These mixed pickles are packed with the following preparation instead of vinegar: Stir  $2\frac{1}{2}$  pounds of best English mustard powder together with 1 pint of best olive oil, and to this add  $2\frac{1}{2}$  lbs. of sugar,  $\frac{1}{2}$  lb. of salt, and 3 to 4 pints of good vinegar, with  $\frac{1}{2}$  pint of curry vinegar. The curry vinegar is prepared from turmeric  $3\frac{1}{2}$  ozs., coriander 4 ozs., black pepper 1 oz., ginger 1 oz., cardamoms  $\frac{1}{2}$  oz., carraway seeds 1 oz., cayenne pepper  $\frac{1}{2}$  oz., all powdered, with  $2\frac{1}{2}$  gallons of wine vinegar. When all has been thoroughly mixed together, the sauce is strained through a hair sieve.

*Pickled Gherkins.*—Cut off the stalks and sort the gherkins into three sizes. Then wash them carefully and pack them into suitable clean barrels. Dill may be used as spice but

nothing else. Pour over them a pickle consisting of 12 per cent. brine containing 25 per cent. of good wine vinegar. Place the barrels in the sun or a warm place, with the bung-holes open, so as to quickly start a slight fermentation. When this has run its course, clean up the barrels, fill them to the top with the same liquor, if necessary, and bung them down. For use the gherkins are taken out of the barrels and packed with fresh wine vinegar spiced with tarragon. If these small gherkins are to be kept stored in the barrels for a long time, the fermented pickle should be drawn off and replaced by fresh 12 per cent. brine.

*Gherkins in Mustard.*—Skin and cut in halves large, ripe gherkins, remove the cores, and then cut them into long strips. Place the latter in tubs, sprinkle well with salt and allow to stand for several days. Then wash the gherkins, pack them into barrels with yellow mustard seed, a few laurel leaves and white pepper-corns, small onions, sliced horse-radish and shredded ginger. Pour in sufficient boiling wine vinegar to cover the whole. After a few days draw off the vinegar, boil it up and return it to the barrels and heavily weight the latter. The vinegar should not be allowed to fall below 4 per cent., otherwise it will have to be supplemented. In 3 to 4 weeks the gherkins should be clear and translucent.

*Pickled Mushrooms.*—Clean and trim off at the bottom young small mushrooms and free them from the brown skin. Then wash them in cold water and carefully dry them. Then pack them in bottles and pour boiling wine vinegar over them.

*Pickled Onions.*—Small onions are peeled and hot vinegar is poured over them. Sometimes the onions are placed in brine for one day.

*Pickled Peaches.*—The fruits, not entirely ripe, are treated like cucumbers.

*Pickled Peas*—The peas are treated like beans and cauliflower.

*Pickled Tomatoes*—must not be entirely ripe; they may be even green and half grown. They are pickled in the same manner as cucumbers.

*Pickled Walnuts.*—Place the young, soft nuts in strong brine for one week, then dry upon a sieve and pour hot vinegar over them. A better method is to expose the walnuts after they have been in the brine for nine days, upon a cloth, to the sun until they are black, and then put them into bottles, which are filled up with hot vinegar. These pickles are much liked with fish, and when well sealed and stored in a dark place keep for ten years. If they are to be used in the first three months after being made, the brine must be heated for one hour.

*Mustard.*—Mustard of commerce is the seed, whole or ground, of several species of the genus *Brassica*, cruciferous plants, which grow wild and are cultivated under various conditions. The two common varieties are the black or brown mustard, which has a very small seed, and furnishes the most aroma, and the white, which is two or three times as large, often used in the whole condition in pickles and ground, either by itself or oftener in mixture with the brown seed, for the purpose of obtaining the desirable qualities of both.

The most rational manner of preparing mustard for table use has been introduced into the English factories. The seed is freed from the husk, ground to flour, and the fat oil, which can be used as an illuminating oil, pressed out. Generally speaking, the preparation of mustard consists in several times grinding in a mill a mixture of white and brown mustard with an addition of wine-must, either fresh or strongly boiled down, or of wine vinegar, until it forms a moderately fine or very fine pasty mass, and adding different substances as a seasoning. In the Düsseldorf mustard the seasoning consists of cinnamon, cloves, and sugar; in the Frankfort mustard of cloves, allspice, and sugar; in the English mustard, of wheat flour, common salt and pepper; and in French mustard, of tarragon, ginger, cinnamon, thyme, marjoram, onions, garlic, cloves, etc. An addition of flour is most generally made, as it modifies the sharpness of the mustard and holds the mass better together. The quantity of the constituents varies, the



usual proportions being from 20 to 30 per cent. of white, and 5 to 10 per cent. of brown, mustard, 1 to 2 per cent. of common salt,  $\frac{1}{4}$  to  $\frac{1}{2}$  per cent. of pulverized spices, and 40 to 50 per cent. of must or vinegar. According to the English method the use of mustard-seed freed from oil is recommended. In the following a few special receipts are given :

*Gumpoldskirchner Must-mustard*.—Evaporate 30 quarts of freshly pressed wine-must to one-half its volume over a moderate fire, dissolve in it 5 lbs. of sugar, and strain the whole over 2 or 3 roots of horseradish cut in thin slices. Then add in the form of fine powder, cardamoms 0.35 oz., nutmeg 0.35 oz., cloves 0.63 oz., cinnamon 1 oz., ginger 1 oz., mustard-seed, ground and freed from oil, brown 6 lbs. and white 11 lbs. Grind the whole several times in a mill and strain.

*Moutard des Jesuites*.—Make a paste of 12 sardines and 280 capers and stir it into 53 ozs. of boiling vinegar, and mix with it ground mustard-seed freed from oil, brown  $5\frac{1}{2}$  ozs. and white  $14\frac{1}{2}$  ounces.

*French Mustard*.—Ground mustard 2 lbs., and  $\frac{1}{2}$  oz. each of fresh parsley and tarragon, both cut up fine, are thoroughly mixed together; further 1 clove of garlic, also add, cut up very fine, 12 salted anchovies. Grind the mixture very fine, add the required must and 1 oz. of pulverized common salt, and for further grinding dilute with water. To evaporate the water after grinding the mustard, heat an iron red-hot and cool it off in the mixture, and then add wine-vinegar of the best quality.

*Ordinary Mustard*.—I. Stir gradually 1 pint of good white wine into 8 ozs. of ground mustard-seed, add a pinch of pulverized cloves, and let the whole boil over a moderate fire. Then add a small lump of white sugar, and let the mixture boil up at once.

II. Pour  $\frac{1}{2}$  pint of boiling wine-vinegar over 8 ozs. of ground mustard-seed in an earthen pot, stir the mixture thoroughly, then add some cold vinegar, and let the pot stand over night in a warm place. The next morning add  $\frac{1}{2}$  lb. of sugar,  $\frac{3}{4}$

drachm of pulverized cinnamon,  $\frac{1}{2}$  drachm of pulverized cloves,  $1\frac{1}{4}$  drachms of pepper, some cardamom and nutmeg, half the rind of a lemon and the necessary quantity of vinegar. The mustard is now ready, and is kept in pots tied up with bladder.

III. Pound to a paste in a mortar the flesh of a salt herring, and 2 ozs. of capers, and mix this with 2 ozs. of pulverized white sugar and 13 ozs. of ground mustard-seed; then pour  $1\frac{3}{4}$  pints of boiling wine vinegar over it, stir, and let the whole stand near a fire for several hours. Finally add  $\frac{3}{4}$  pint of boiling vinegar, stir thoroughly and pour the mustard into glass bottles.

*Frankfort Mustard.*—Mix 1 lb. of white mustard-seed, ground, a like quantity of brown mustard-seed, 8 ozs. of pulverized sugar, 1 oz. of pulverized cloves, 2 ozs. of allspice, and compound the mixture with white-wine or wine-vinegar.

*Wine Mustard.*—Ground mustard-seed, white, 23 ozs., brown 12 ozs., common salt  $2\frac{3}{4}$  ozs., wine vinegar  $8\frac{1}{2}$  ozs., a like quantity of white wine, and water 16 ozs.

*Aromatic or Hygienic Mustard.*—Ground mustard-seed, white, 23 ozs., brown 12 ozs., wine vinegar  $17\frac{1}{2}$  ozs. Extract allspice 0.35 oz., cassia, white pepper, and ginger, of each 0.17 oz., with alcohol  $1\frac{1}{2}$  ozs., and water  $8\frac{1}{2}$  ozs., add  $3\frac{1}{2}$  ozs. of common salt and a like quantity of sugar, filter the whole and add it to the mustard.

*Düsseldorf Mustard.*—Ground mustard-seed freed from oil, brown 3 ozs., white  $8\frac{1}{2}$  ozs., boiling water  $26\frac{1}{2}$  ozs., wine vinegar 18 ozs., cinnamon 0.17 oz., cloves 0.1 oz., sugar 11 ozs., white wine 18 ozs.

*Sour Düsseldorf Mustard.*—Fill 2 casks with vinegar, steep in one of the casks 2 lbs. of organ leaves, and in the other an ordinary bucket full of onions cut up, and let them digest for 2 days. Then bruise 44 lbs. of white mustard-seed and 66 lbs. of brown; put this in a vat and add 1 lb. of pulverized cloves,  $1\frac{1}{2}$  lbs. of pulverized coriander seed, and  $4\frac{1}{2}$  gallons of each of the prepared vinegars. Stir the whole thor-

oughly and grind it twice in a mill. To every gallon of this add and mix thoroughly with it, 1 lb. of salt dissolved in 1 quart of the onion vinegar.

*Sweet Kremser Must-mustard.*—Ground mustard-seed, brown 10 lbs., white 5 lbs., is intimately mixed with 3 lbs. of freshly-pressed must, and boiled down to the desired consistency.

*Sour Kremser Must-mustard.*—Boil to a stiff paste 15 lbs. of brown mustard ground, and 5 lbs. of white mustard ground, together with 4 lbs. of must, and after cooling stir in 4 lbs. of vinegar.

*Moutarde de maille.*—Cut up 8 ozs. of fresh tarragon leaves without the stems,  $2\frac{1}{4}$  ozs. of basil, 2 ozs. of bay leaves and 4 ozs. of rocambole (a spice of garlic). Place these ingredients in a glass alembic, pour  $2\frac{1}{2}$  quarts of strong wine-vinegar over them, and, to allow the vapors to escape, tie up the mouth of the alembic with a piece of perforated moist bladder. Place the alembic upon hot sand for 4 days, then filter the fluid first through linen and then through blotting paper. Add to this aromatic vinegar, 1 oz. of common salt, then stir it into a thick paste with ground brown mustard-seed, and keep the the mustard in earthenware jars tied up with bladder.

*Moutarde aux épices* is prepared by extracting 18 ozs. of tarragon leaves, 7 ozs. of basil,  $1\frac{3}{4}$  ozs. of bay leaves,  $3\frac{1}{2}$  ozs. of white pepper,  $1\frac{3}{4}$  ozs. of cloves, and 0.35 oz. of mace with vinegar and mixing the extract with mustard prepared in the ordinary manner from ground mustard-seed, brown 44 lbs., white 11 lbs., and vinegar  $8\frac{1}{2}$  lbs.

*Moutarde Aromatisée.*—Boil ground mustard-seed, brown 22 lbs., white 44 lbs, with 9 lbs. of vinegar, and add oil of tarragon 1 oz., oil of thyme  $\frac{1}{2}$  oz., oil of mace 0.35 oz., and oil of cloves 0.17 oz., all previously dissolved in very strong vinegar.

*English Mustard.*—Ground mustard-seed 9 lbs., wheat flour 9 ozs., common salt  $1\frac{3}{4}$  lb., cayenne pepper  $2\frac{3}{4}$  ozs., and as much vinegar and water as required.



## CHAPTER XXXIV.

## PRESERVATION OF MEAT, FISH AND EGGS.

*Appert's Method of Canning Meats.*—This method consists in soldering up the food in tin cans with great care, and then heating the cans for considerable time in water. The cans used are either cylinders or four-sided cases. Although the latter do not waste so much space as the former when packed with others, the cylinders are generally preferred, they being easier to make by machinery and much easier to solder up. The duration of the heating depends on the size of the can, for every part of its contents must be kept at the temperature of boiling water for a sufficient time. Meat is a bad conductor of heat and it takes considerable time before the contents of larger cans are heated through and through to 212° F.

The time required for the purpose can only be learned by experience. Insert in one of the cans a thermometer so that the bulb is exactly in the centre of the can, and observe repeatedly the time which elapses between when the water commences to boil and the moment at which the mercury rises to 212° F.

In placing the prepared meats in the cans, the pieces should be so arranged as to leave but few interspaces between them, and the cans should be filled as full as possible, so that but little air remains in them. Immediately after the cans are filled, the lids are soldered on. When a sufficient number of cans have been soldered, they are at once heated to the temperature of boiling water. Heating by steam is more suitable and less troublesome than by water. For this purpose the cans are so arranged in a chamber that the steam can circulate freely round each one, the bottom layer of cans resting on a lattice bottom. The steam enters the chamber below this lattice bottom and escapes through an aperture on the upper side of the chamber. Below the lattice bottom is a trap for drawing off the condensed water. At first the steam

is admitted at a pressure of two to three atmospheres. The steam condenses on the cold cans and, when condensation has ceased, the blow-off cock is shut, and the passage of steam is continued until the contents of the cans have been heated to 212° F. The time required for this purpose depends on the size of the chamber, the number of cans in it, the quantity and temperature of the steam introduced, etc. In all cases, whether heating is done by water or by steam, care must be had to only gradually increase the heat to give the heated oxygen time to be absorbed by the meat. By heating too rapidly it may happen that, in consequence of the great pressure exerted by the strongly heated air upon the sides of the cans, the soldered places may become defective, and the cans be spoiled.

When heating has been successfully finished, *i. e.*, when all the oxygen has been absorbed and the cans remain perfectly tight, it will frequently be observed that, after cooling, the bottoms of the cans bulge inward, though they were perfectly flat previous to heating. This is a favorable indication of successful preservation of the contents, because the oxygen contained in the box being completely fixed to the meat, the tension of the other enclosed gas (nitrogen) is considerably decreased in cooling, which causes the slight bulging inward of the bottoms. When, on the other hand, it is observed that the bottoms of cans which have been stored for some time bulge outward, it is an indication that the contents are in a state of decomposition produced by the ferments contained in them not having been destroyed. In consequence of decomposition gases are evolved which accumulate more and more, and by their pressure force the bottoms to bulge outward and finally burst the cans.

The object to be attained in operating according to Appert's method, is to leave as little air as possible in the cans. This object may be reached in various ways. With meat, which is to be canned together with the broth, the smaller cans are closed in the usual manner by soldering, and a small hole is

punched in the lids. When the cans have been sufficiently heated and the steam escapes from the small holes, the latter are closed by a drop of solder. For larger cans sufficient water is added to the broth to fill the cans nearly to the rims. They are then closed in the usual manner, a small hole is punched in the lids and the cans are heated as long as a current of steam escapes from the holes, when the latter are closed by a drop of solder. In consequence of heating, the expanded air escapes first from the cans, the last portions being displaced by the steam. After closing the small holes and cooling, the free spaces in the cans are almost free from air. When a hole is punched in a can a peculiar hissing sound is heard, which is produced by the air rushing into the can.

Appert's method is adapted for all kinds of meat, whether stewed or roasted, with or without broth. To save space the bones of larger animals are removed and the cans are packed with pieces of meat. Smaller animals, for instance, poultry, are cut up and then canned.

*Corned beef* is prepared by Appert's method. The meat is freed from fat and cut into slices. The latter are then packed closely interstratified with a little salt into cans of a truncated pyramidal shape. The cooking is done with high-pressure steam, and causes the slices to cohere into a solid mass.

*Meat Biscuit according to Gallamond.*—The preparation of this product embraces three operations: Preparation of the bouillon, preparation of the dough, and baking of the biscuit.

1. *Preparation of the bouillon.* Bring 56 lbs. of beef and 24 quarts of water in a boiler, add, tied in a linen bag, thyme, bay leaves, two nutmegs, cloves, pepper, cinnamon or ginger, and 22 lbs. of vegetables (carrots, turnips, leek). After boiling for four hours, remove the bones from the meat, divide the latter into small pieces, return them to the bouillon and continue boiling for  $1\frac{1}{2}$  hours longer. The contents of the boiler are then of the consistency of a very thin paste. Dissolve in this paste about  $\frac{1}{2}$  lb. of rock candy, which is claimed to increase the keeping qualities of the biscuit. In the manner



above described about 11 quarts of very concentrated broth are obtained, which contains all the soluble parts and the fibrin of 48 lbs. of meat (8 lbs. being lost in the form of bones, sinews, etc.).

2. *Preparation of the dough.* 110 lbs. of wheat flour are incorporated by kneading with the 11 quarts of bouillon. The dough is very solid and is cut up into biscuits, generally 237.

3. *Baking the Biscuit.* The biscuits remain in the oven for  $1\frac{1}{2}$  hours. The composition of 100 parts of this biscuit is about as follows: Dry flour, 76.45; dry meat, 5.79; fat, 6.27; dry vegetables, 2.77; spices and sugar, 0.92; water, 7.80.

*Soup Tablets.* For the preparation of soup tablets it is first of all necessary to extract the soluble matters from the meat. The proper plan to do this is to keep the meat in cold water and then to heat it gradually and not to a temperature so high as to coagulate the albumen. If the meat is plunged from the start in boiling water, the outside of it is made impervious by the coagulation of the albumen and the soluble constituents are only imperfectly extracted even by long boiling. The meat should be scrupulously cleaned from fat. It is then minced in a mincing machine or cut up small with a chopper, and steeped in water which should not be too hard and is gradually brought to a temperature not exceeding  $140^{\circ}\text{F}$ . Heating is kept up, whilst constantly stirring, for several hours. When working on a large scale a mechanical stirrer should be used. The proportion of water to meat should be two parts to one by weight. After heating for three hours nothing will be left undissolved but worthless fibrin. The temperature is then raised to about  $194^{\circ}\text{F}$ . The liquid, previously clear, then becomes thick, and a scum rises to the surface, both these appearances being due to the coagulation of the albumen. The liquid thus prepared may be stored by bringing it, while still hot, into vessels, filling them quite full and closing them air-tight. The liquid will keep for many months.

For the preparation of soup tablets, the liquid is mixed with

about 1 lb. of salt for every 100 lbs. of meat used and filtered through several folds of linen. The filtrate is evaporated in very shallow tin-plate pans, without, however, allowing it to boil, till a sample becomes hard when cold. The entire mass is then cast into molds. When properly made soup tablets are usually of a pale brown color and give a perfectly clear solution in water, having the taste of fresh beef tea.

*Beef extract.*—This product is made at Fray Bentos in Uruguay, by the following method. The meat from the cattle of the grass plains of the pampas is freed from bones and fat, and minced. The paste is mixed with water and subjected to great hydraulic pressure. The concentrated solution of the soluble constituents of the meat thus obtained is at once boiled to coagulate the albumen. The clear liquid is then evaporated in vacuum pans till the mass on cooling is of a semi-solid consistency. When prepared in this manner the extract contains no gelatine and forms a perfectly clear solution with water. The absence of gelatine and fat makes the extract so unalterable that a pot of it can be left for as long as desired in a damp mouldy room without undergoing change.

*Quick Salting of Meat by liquid pressure.*—This process is especially applicable to the preservation of large masses. A reservoir of concentrated brine is placed on a high level, at least 30 feet above the room where the process is worked. A pipe brought down from the reservoir is connected by a length of rubber tube with an iron-pointed pipe. This pipe is about 8 inches long, is provided with a tap and has numerous holes bored in it towards the point. If this point is inserted into the middle of a piece of meat and the tap is opened, the hydrostatic pressure causes the brine to penetrate the meat completely in a very few minutes. The pieces impregnated in this manner are placed for about 24 hours in concentrated brine, and the meat loses no nutritive matter as it is already thoroughly penetrated with brine. The pieces are then simply dried or slightly smoked.

*Quick Process of Smoking Meat.*—This process is based upon

the fact that crude wood vinegar (pyroligneous acid) as obtained in the destructive distillation of wood contains creosote as well as acetic acid and water. It has a strong smoky smell and is a powerful antiseptic. The meat is for a few seconds dipped in the fluid, allowed to drain off and then hung up to dry. The room in which the work is done should be warm enough for the fat of the meat to become soft, the absorption of the wood vinegar being thereby facilitated. When the meat has absorbed the acid it is again dipped two or three times in the wood vinegar, and then left to itself for 48 to 60 hours when it may be considered as thoroughly cured. To give the cured meat the aromatic taste of juniper, a small quantity of oil of juniper is dissolved in the wood vinegar diluted with water. A fluid of the following composition yields excellent results :

Crude wood vinegar . . . . .	50 gallons.
Water . . . . .	100 “
Oil of juniper . . . . .	2½ “

Although the acetic acid contained in the wood vinegar acts as a preservative, the greater portion of it is lost by evaporation, and hence a fluid may suitably be used which contains only a small quantity of wood vinegar, but a larger quantity of the actually effective creosote. Such a fluid may be prepared as follows :

Water . . . . .	500 parts by weight.
Creosote . . . . .	5 “ “
Crude wood vinegar . . . . .	50 “ “
Oil of juniper . . . . .	5 “ “

Dissolve the creosote in the wood vinegar, dissolve in this solution the oil of juniper, and pour the fluid in a thin jet, with constant stirring, into the water.

Curing may also be very rapidly effected by bringing the meat into a smoke-house connected with a well-drawing chimney, and placing on the floor of the smoke-house shallow dishes containing a mixture of



Wood vinegar . . . . .	50 parts.
Creosote . . . . .	10 "
Water. . . . .	50 "

Place a large bath sponge in each dish, and conduct a current of warm air over the dishes.

By the current of warm air the fluid absorbed by the sponges is evaporated, and curing rapidly effected.

*Preparation of Powdered Meat.*—An excellent product—a kind of pemmican—is obtained by a process patented by Hassal. The meat used for the purpose should be as free as possible from fat, veins and sinews, fat being particularly subject to decomposition. The meat is cut up in a mincing machine to a paste, and the latter is spread out in thin layers on tin plate and dried in a drying room, a very convenient way being to force hot air over the layers by means of a fan. The temperature should never exceed 140° F., otherwise the albuminous constituents of the meat coagulate and become insoluble. Drying is continued at between 122° and 140° F. till the meat is considerably reduced in volume and forms a crusty mass. The latter is next reduced to powder in a mill, dried again, and then brought into metal cans or paper cylinders lined with tin-foil. A soup of excellent taste and quality is obtained from this meat powder by simmering for some time in water at between 122° and 140° F.

*Preservation of Fish.*—In order to obtain a good product, fish which are to be preserved in oil should be treated a few hours after having been caught. The fish should be carefully cleaned, the entrails taken out, the tails cut off, and eventually the heads also, as they impart a bitter taste to the fish. Finally the fish are for a short time laid in brine. They are then taken out, thoroughly rinsed in clean water, laid upon grates to drain, and finally dried in the air, or, when this is impossible on account of unfavorable weather, in ovens of special construction, in which they remain until they feel perfectly dry and solid. They are then laid upon shallow wire sieves, and boiled in olive oil until done. The boiler is generally heated by steam.

The oil should have a temperature of  $310^{\circ}$  to  $338^{\circ}$  F., and, according to the size of the fish, 45 to 80 seconds are required for boiling. When boiling is finished, the sieves with the fish are taken from the oil, and after draining off and drying, the fish are packed, according to sizes, in tin cans. The latter are then filled with oil, closed by soldering on the lids, and, according to Appert's method, again boiled. Separate pieces of larger fish are treated in the same manner.

According to Appert's method, all kinds of fish, lobster, crabs, oysters, etc., can be preserved by preparing them in the same manner as for immediate consumption, then putting them in tin cans, closing the latter by soldering, and heating them in hot water or by steam, so that the contents are exposed to a temperature of at least  $212^{\circ}$  F.

*Preservation of Eggs.*—Eggs are frequently preserved by means of lime water, which, to be sure, is one of the most convenient, but at the same time one of the worst methods, the eggs acquiring the disagreeable taste of lime.

To prevent eggs preserved in lime water from acquiring a disagreeable taste of lime, Kubel recommends lime water of 1.0029 specific gravity, and to dissolve in it 6 per cent. of common salt, so that the fluid shows a specific gravity of 1.042. Perhaps a 6 per cent. common salt solution without lime water would answer as well.

A good preserving fluid is commercial glycerin diluted with half its volume of well water. Salicylic acid is also very useful for the purpose. Prepare a mixture of 2 quarts of water and 1 quart strong alcohol, add 7 ozs. of glycerin, and dissolve in this fluid as much crystallized salicylic acid as will dissolve in it. Bring the eggs to be preserved into the fluid, allow them to remain for one hour, and then let them dry, placing them for this purpose in a frame of boards or sheet zinc provided with holes, one egg being placed in each hole. In drying, the water and alcohol rapidly evaporate, and the salicylic acid dissolved in them would separate in crystals upon the surface of the eggs. However, the glycerin does not evaporate, as it

possesses the property of vigorously absorbing water from the air, and, besides, dissolves salicylic acid. Hence a fluid is formed which consists of a saturated solution of salicylic acid in glycerin and fills the pores of the eggs.

The expense of preserving eggs by this method is very small, since a fluid containing 2 lbs. of salicylic acid in solution suffices for many thousands of eggs. In order not to waste any of the preserving fluid, the drippings from the eggs already treated may be collected in a vessel and again used.

Eggs preserved in this manner may be kept for many months at the ordinary temperature of living-rooms without the slightest alteration.

The result of the competition for prizes offered by the Association of Poultry Breeders of the Province of Saxony, Prussia, has shown that water-glass (silicate of soda) is an excellent preservative for eggs. The first prize was awarded to a competitor who had used the following process: The eggs, which should be perfectly clean, were placed in a can filled with a 10 per cent. solution of silicate of soda and the can was closed air-tight. The solution was once renewed during the preserving time of six months. The temperature of the storage room on the hottest day was 77° F. In appearance the eggs could not be distinguished from fresh eggs. The yolk and white were perfect, and the taste excellent.

A simple method of preserving eggs by means of silicate of soda is as follows: Immerse the eggs in a solution of silicate of soda for about 10 minutes by floating a board on top of them. Then remove them and stand them up with their small ends in little holes in a shelf for two days, when they can be packed for storage or transit. The silica of the silicate of soda is set free by the carbon dioxide of the air and then forms with the lime of the egg-shell a glassy sheet of calcium silicate which closes all the pores.

The competition previously referred to has further shown that the breed of chickens is without influence upon the preserving capacity of the eggs, which, however, appears some-



what affected by the feed. As is well known, eggs of chickens which run free and subsist chiefly on animal food—worms, insects, etc.—have a reddish-yellow yolk, while chickens fed chiefly with grain lay eggs with a pale yellow yolk. The latter have a finer taste than the former. Exporters also assert that eggs with a red-yellow yolk do not keep so well, and in Russia, for instance, are for this reason excluded as much as possible from export. To recognize the color of the yolk, hold the egg before the light.

# APPENDIX.

TABLE I.—*Hegner's Alcohol Table.*

Specific gravity at 60° F.	Per cent. by weight of absolute alcohol.	Per cent. by volume of absolute alcohol.	Specific gravity at 60° F.	Per cent. by weight of absolute alcohol.	Per cent. by volume of absolute alcohol.	Specific gravity at 60° F.	Per cent. by weight of absolute alcohol.	Per cent. by volume of absolute alcohol.	Specific gravity at 60° F.	Per cent. by weight of absolute alcohol.	Per cent. by volume of absolute alcohol.
1.0000	0.00	0.00	0.9969	1.75	2.20	0.9938	3.53	4.42	0.9907	5.44	6.78
			8	1.81	2.27	7	3.59	4.49	6	5.50	6.86
0.9999	0.05	0.07	7	1.87	2.35	6	3.65	4.56	5	5.56	6.94
8	0.11	0.13	6	1.94	2.43	5	3.71	4.63	4	5.62	7.01
7	0.16	0.20	5	2.00	2.51	4	3.76	4.71	3	5.69	7.09
6	0.21	0.26	4	2.06	2.58	3	3.82	4.78	2	5.75	7.17
5	0.26	0.32	3	2.11	2.65	2	3.88	4.85	1	5.81	7.25
4	0.32	0.40	2	2.17	2.72	1	3.94	4.93	0	5.87	7.32
3	0.37	0.46	1	2.22	2.79	0	4.00	5.00			
2	0.42	0.53	0	2.28	2.86				0.9899	5.94	7.40
1	0.47	0.60				0.9929	4.06	5.08	8	6.00	7.48
0	0.53	0.66	0.9959	2.33	2.93	8	4.12	5.16	7	6.07	7.57
			8	2.39	3.00	7	4.19	5.24	6	6.14	7.66
0.9989	0.58	0.73	7	2.45	3.07	6	4.25	5.32	5	6.21	7.74
8	0.63	0.79	6	2.51	3.14	5	4.31	5.39	4	6.28	7.83
7	0.68	0.86	5	2.57	3.21	4	4.37	5.47	3	6.36	7.92
6	0.74	0.93	4	2.61	3.28	3	4.44	5.55	2	6.43	8.01
5	0.79	0.99	3	2.65	3.35	2	4.50	5.63	1	6.50	8.10
4	0.84	1.06	2	2.71	3.42	1	4.56	5.71	0	6.57	8.18
3	0.89	1.13	1	2.78	3.49	0	4.62	5.78			
2	0.95	1.19	0	2.84	3.55				0.9889	6.64	8.27
1	1.00	1.26				0.9919	4.69	5.86	8	6.71	8.36
0	1.06	1.34	0.9949	2.89	3.62	8	4.75	5.94	7	6.78	8.45
			8	2.94	3.69	7	4.81	6.02	6	6.86	8.54
0.9979	1.12	1.42	7	3.00	3.76	6	4.87	6.10	5	6.93	8.63
8	1.19	1.49	6	3.06	3.83	5	4.94	6.17	4	7.00	8.72
7	1.25	1.57	5	3.12	3.90	4	5.00	6.24	3	7.06	8.80
6	1.31	1.65	4	3.18	3.98	3	5.06	6.32	2	7.13	8.88
5	1.37	1.73	3	3.24	4.05	2	5.12	6.40	1	7.19	8.96
4	1.44	1.81	2	3.29	4.12	1	5.19	6.48	0	7.27	9.04
3	1.50	1.88	1	3.35	4.20	0	5.25	6.55			
2	1.56	1.96	0	3.41	4.27				0.9879	7.33	9.13
1	1.62	2.04				0.9909	5.31	6.63	8	7.40	9.21
0	1.69	2.12	0.9939	3.47	4.34	8	5.37	6.71	7	7.47	9.29

TABLE I.—*Continued.*

Specific gravity at 60° F.	Per cent. by weight of absolute alcohol.	Per cent. by volume of absolute alcohol.	Specific gravity at 60° F.	Per cent. by weight of absolute alcohol.	Per cent. by volume of absolute alcohol.	Specific gravity at 60° F.	Per cent. by weight of absolute alcohol.	Per cent. by volume of absolute alcohol.	Specific gravity at 60° F.	Per cent. by weight of absolute alcohol.	Per cent. by volume of absolute alcohol.
0.9876	7.53	9.37	0.9836	10.38	12.87	0.9796	13.46	16.61	0.9756	16.77	20.61
5	7.60	9.45	5	10.46	12.96	5	13.54	16.70	5	16.85	20.71
4	7.67	9.54	4	10.54	13.05	4	13.62	16.80	4	16.92	20.80
3	7.73	9.62	3	10.62	13.15	3	13.69	16.89	3	17.00	20.89
2	7.80	9.70	2	10.69	13.24	2	13.77	16.98	2	17.08	20.99
1	7.87	9.78	1	10.77	13.34	1	13.85	17.08	1	17.17	21.09
0	7.93	9.86	0	10.85	13.43	0	13.92	17.17	0	17.25	21.19
0.9869	8.00	9.95	0.9829	10.92	13.52	0.9789	14.00	17.26	0.9749	17.33	21.29
8	8.07	10.03	8	11.00	13.62	8	14.09	17.37	8	17.42	21.39
7	8.14	10.12	7	11.08	13.71	7	14.18	17.48	7	17.50	21.49
6	8.21	10.21	6	11.15	13.81	6	14.27	17.59	6	17.58	21.59
5	8.29	10.30	5	11.23	13.90	5	14.36	17.70	5	17.67	21.69
4	8.36	10.38	4	11.31	13.99	4	14.45	17.81	4	17.75	21.79
3	8.43	10.47	3	11.38	14.09	3	14.55	17.92	3	17.83	21.89
2	8.50	10.56	2	11.46	14.18	2	14.64	18.03	2	17.92	21.99
1	8.57	10.65	1	11.54	14.27	1	14.73	18.14	1	18.00	22.09
0	8.64	10.73	0	11.62	14.37	0	14.82	18.25	0	18.08	22.18
0.9859	8.71	10.82	0.9819	11.69	14.46	0.9779	14.91	18.36	0.9739	18.15	22.27
8	8.79	10.91	8	11.77	14.56	8	15.00	18.48	8	18.23	22.36
7	8.86	11.00	7	11.85	14.65	7	15.08	18.58	7	18.31	22.46
6	8.93	11.08	6	11.92	14.74	6	15.17	18.68	6	18.38	22.55
5	9.00	11.17	5	12.00	14.84	5	15.25	18.78	5	18.46	22.64
4	9.07	11.26	4	12.08	14.93	4	15.33	18.88	4	18.54	22.73
3	9.14	11.35	3	12.15	15.02	3	15.42	18.98	3	18.62	22.82
2	9.21	11.44	2	12.23	15.12	2	15.50	19.08	2	18.69	22.92
1	9.29	11.52	1	12.31	15.21	1	15.58	19.18	1	18.77	23.01
0	9.36	11.61	0	12.38	15.30	0	15.67	19.28	0	18.85	23.10
0.9849	9.43	11.70	0.9809	12.46	15.40	0.9769	15.75	19.39	0.9729	18.92	23.19
8	9.50	11.79	8	12.54	15.49	8	15.83	19.49	8	19.00	23.28
7	9.57	11.87	7	12.62	15.58	7	15.92	19.59	7	19.08	23.38
6	9.64	11.96	6	12.69	15.68	6	16.00	19.68	6	19.17	23.48
5	9.71	12.05	5	12.77	15.77	5	16.08	19.78	5	19.25	23.58
4	9.79	12.13	4	12.85	15.86	4	16.15	19.87	4	19.33	23.68
3	9.86	12.22	3	12.92	15.96	3	16.23	19.96	3	19.42	23.78
2	9.93	12.31	2	13.00	16.05	2	16.31	20.06	2	19.50	23.88
1	10.00	12.40	1	13.08	16.15	1	16.38	20.15	1	19.58	23.98
0	10.08	12.49	0	13.15	16.24	0	16.46	20.24	0	19.67	24.08
0.9839	10.15	12.58	0.9799	13.23	16.33	0.9759	16.54	20.33	0.9719	19.75	24.18
8	10.23	12.68	8	13.31	16.43	8	16.62	20.43	8	19.83	24.28
7	10.31	12.77	7	13.38	16.52	7	16.69	20.52	7	19.92	24.38



TABLE I.—*Concluded.*

Specific gravity at 60° F.	Per cent. by weight of absolute alcohol.	Per cent by volume of absolute alcohol.	Specific gravity at 60° F.	Per cent. by weight of absolute alcohol.	Per cent. by volume of absolute alcohol.	Specific gravity at 60° F.	Per cent. by weight of absolute alcohol.	Per cent. by volume of absolute alcohol.	Specific gravity at 60° F.	Per cent. by weight of absolute alcohol.	Per cent. by volume of absolute alcohol.
0.9716	20.00	24.48	0.9699	21.38	26.13	0.9682	22.69	27.68	0.9666	23.92	29.13
5	20.08	24.58	8	21.46	26.22	1	22.77	27.77	5	24.00	29.22
4	20.17	24.68	7	21.54	26.31	0	22.85	27.86	4	24.08	29.31
3	20.25	24.78	6	21.62	26.40				3	24.15	29.40
2	20.33	24.88	5	21.69	26.49	0.9679	22.92	27.95	2	24.23	29.49
1	20.42	24.98	4	21.77	26.58	8	23.00	28.04	1	24.31	29.58
0	20.50	25.07	3	21.85	26.67	7	23.08	28.13	0	24.38	29.67
			2	21.92	26.77	6	23.15	28.22			
0.9709	20.58	25.17	1	22.00	26.86	5	23.23	28.31	0.9659	24.46	29.76
8	20.67	25.27	0	22.08	26.95	4	23.31	28.41	8	24.54	29.86
7	20.75	25.37				3	23.38	28.50	7	24.62	29.95
6	20.83	25.47	0.9689	22.15	27.04	2	23.46	28.59	6	24.69	30.04
5	20.92	25.57	8	22.23	27.13	1	23.54	28.68	5	24.77	30.13
4	21.00	25.67	7	22.31	27.22	0	23.62	28.77	4	24.85	30.22
3	21.08	25.76	6	22.38	27.31				3	24.92	30.31
2	21.15	25.86	5	22.46	27.40	0.9669	23.69	28.86	2	25.00	30.40
1	21.23	25.95	4	22.54	27.49	8	23.77	28.95			
0	21.31	26.04	3	22.62	27.59	7	23.85	29.04			

TABLE II.—Which indicates the specific gravity of mixtures of alcohol and water.

The figures in the column to the left show the per cent. by volume of alcohol; the figures in the column to the right give the specific gravities which correspond to the content of alcohol at 60° F.

Per cent. by volume of alcohol.	Specific gravity at 60° F.	Per cent. by volume of alcohol.	Specific gravity at 60° F.	Per cent. by volume of alcohol.	Specific gravity at 60° F.	Per cent. by volume of alcohol.	Specific gravity at 60° F.
1	0.9985	26	0.9698	51	0.9323	76	0.8747
2	0.9970	27	0.9688	52	0.9303	77	0.8720
3	0.9956	28	0.9677	53	0.9283	78	0.8693
4	0.9942	29	0.9666	54	0.9263	79	0.8665
5	0.9928	30	0.9655	55	0.9242	80	0.8639
6	0.9915	31	0.9643	56	0.9221	81	0.8611
7	0.9902	32	0.9631	57	0.9200	82	0.8583
8	0.9880	33	0.9618	58	0.9178	83	0.8555
9	0.9878	34	0.9605	59	0.9156	84	0.8526
10	0.9866	35	0.9592	60	0.9134	85	0.8496
11	0.9854	36	0.9579	61	0.9112	86	0.8466
12	0.9843	37	0.9565	62	0.9090	87	0.8436
13	0.9832	38	0.9550	63	0.9067	88	0.8405
14	0.9821	39	0.9535	64	0.9044	89	0.8373
15	0.9812	40	0.9519	65	0.9021	90	0.8339
16	0.9800	41	0.9503	66	0.8997	91	0.8306
17	0.9790	42	0.9487	67	0.8973	92	0.8272
18	0.9780	43	0.9470	68	0.8949	93	0.8237
19	0.9770	44	0.9452	69	0.8925	94	0.8201
20	0.9760	45	0.9435	70	0.8900	95	0.8164
21	0.9750	46	0.9417	71	0.8875	96	0.8125
22	0.9740	47	0.9399	72	0.8850	97	0.8084
23	0.9729	48	0.9381	73	0.8825	98	0.8041
24	0.9719	49	0.9362	74	0.8799	99	0.7995
25	0.9709	50	0.9343	75	0.8773	100	0.7946





TABLE IV.—*The actual content of alcohol and water in mixtures of both fluids, and the contraction which takes place in mixing.*

Specific gravity.	100 volumes contain volumes—		Contraction.	Specific gravity.	100 volumes contain volumes—		Contraction.
	Alcohol.	Water.			Alcohol.	Water.	
1.0000	0	100.000	0.000	0.9323	51	52.705	3.705
0.9985	1	99.055	055	03	52	51.711	711
70	2	98.111	111	0.9283	53	50.716	716
56	3	97.176	176	63	54	49.722	722
42	4	96.242	242	42	55	48.717	717
28	5	95.307	307	21	56	47.712	712
15	6	94.382	382	0.9200	57	46.708	708
02	7	93.458	458	0.9178	58	45.693	693
0.9890	8	92.543	543	56	59	44.678	678
78	9	91.629	629	34	60	43.664	664
66	10	90.714	714	12	61	42.649	649
54	11	89.799	799	0.9090	62	41.635	635
43	12	88.895	895	67	63	40.610	610
32	13	87.990	990	44	64	39.586	586
21	14	87.086	1.086	21	65	38.561	561
11	15	86.191	191	0.8997	66	37.526	526
0.9800	16	85.286	286	73	67	36.492	492
0.9790	17	84.392	392	49	68	35.457	457
80	18	83.497	497	25	69	34.423	423
70	19	82.603	603	0.8900	70	33.378	378
60	20	81.708	708	75	71	32.333	333
50	21	80.813	813	50	72	31.289	289
40	22	79.919	919	25	73	30.244	244
29	23	79.014	2.014	0.8799	74	29.190	190
19	24	78.119	119	73	75	28.135	135
09	25	77.225	225	47	76	27.080	080
0.9698	26	76.320	320	20	77	26.016	016
88	27	75.426	426	0.8693	78	24.951	2.951
77	28	74.521	521	65	79	23.877	877
66	29	73.617	617	39	80	22.822	822
55	30	72.712	712	11	81	21.747	747
43	31	71.797	797	0.8583	82	20.673	673
31	32	70.883	883	55	83	19.598	598
18	33	69.958	958	26	84	18.514	514
05	34	69.034	3.034	0.8496	85	17.419	419
0.9592	35	68.109	109	66	86	16.324	324
79	36	67.184	184	36	87	15.230	230
65	37	66.250	250	05	88	14.125	125
50	38	65.305	305	0.8373	89	13.011	011
35	39	64.361	361	39	90	11.876	1.876
19	40	63.406	406	06	91	10.751	751
03	41	62.451	451	0.8272	92	9.617	617
0.9487	42	61.497	497	37	93	8.472	472
70	43	60.532	532	01	94	7.318	318
52	44	59.558	558	0.8764	95	6.153	153
35	45	58.593	593	25	96	4.968	0.968
17	46	57.618	618	0.8084	97	3.764	764
0.9399	47	56.644	644	41	98	2.539	539
81	48	55.669	669	0.7995	99	1.285	285
62	49	54.685	685	46	100	0.000	000
43	50	53.700	700				

TABLE V.—*For comparing the different aerometers with Tralles's alcoholometer.*

The statements of figures of the other aerometers corresponding to the per cent. by volume according to Tralles's alcoholometer stand in the same horizontal line.

Per cent. by volume according to Tralles.	Per cent. by weight.	Aerometer of—				Per cent. by volume according to Tralles	Per cent. by weight.	Aerometer of—			
		Richter.	Beck.	Beaumé.	Cartier.			Richter.	Beck.	Beaumé.	Cartier.
0	0.	0.0	0.0	10	11	51	43.47	—	12.3	—	—
1	0.80	—	—	—	—	52	44.42	—	12.7	—	—
2	1.60	—	—	—	—	53	45.36	—	13.1	21	—
3	2.40	—	—	—	—	54	46.32	—	13.5	—	21
4	3.20	—	1.0	—	—	55	47.29	41.00	13.9	—	—
5	4.10	4.00	1.2	11	12	56	48.26	—	14.3	22	—
6	4.81	—	1.4	—	—	57	49.23	—	14.8	—	22
7	5.62	—	1.6	—	—	58	50.21	—	15.2	23	—
8	6.43	—	1.9	—	—	59	51.20	—	15.6	—	—
9	7.24	—	2.1	—	—	60	52.20	45.95	16.1	—	23
10	8.05	7.50	2.3	12	—	61	53.20	—	16.5	24	—
11	8.87	—	2.5	—	—	62	54.21	—	17.0	—	—
12	9.69	—	2.7	—	13	63	55.21	—	17.5	25	24
13	10.51	—	2.9	—	—	64	56.22	—	18.0	—	—
14	11.33	—	3.1	—	—	65	57.24	51.40	18.4	—	25
15	12.15	10.58	3.3	—	—	66	58.27	—	18.9	26	—
16	12.98	—	3.5	13	—	67	59.32	—	19.4	—	—
17	13.80	—	3.6	—	—	68	60.38	—	20.0	27	26
18	14.63	—	3.8	—	—	69	61.42	—	20.5	—	—
19	15.46	—	4.0	—	14	70	62.50	57.12	21.0	28	27
20	16.28	13.55	4.2	—	—	71	63.58	—	21.5	—	—
21	17.11	—	4.4	—	—	72	64.66	—	22.1	—	—
22	17.95	—	4.6	—	—	73	65.74	—	22.6	29	28
23	18.78	—	4.8	14	—	74	66.83	—	23.2	—	—
24	19.62	—	4.9	—	—	75	67.93	62.97	23.8	30	29
25	20.46	16.60	5.1	—	—	76	69.05	—	24.4	—	—
26	21.30	—	5.3	—	15	77	70.18	—	25.0	31	30
27	22.14	—	5.5	—	—	78	71.31	—	25.6	—	—
28	22.96	—	5.7	—	—	79	72.45	—	26.2	32	—
29	23.84	—	5.9	15	—	80	73.59	69.20	26.8	—	31
30	24.69	19.78	6.1	—	—	81	74.74	—	27.4	33	—
31	25.55	—	6.4	—	—	82	75.91	—	28.0	34	32
32	26.41	—	6.6	—	—	83	77.09	—	28.7	—	—
33	27.27	—	6.8	—	16	84	78.29	—	29.4	35	33
34	28.13	—	7.0	16	—	85	79.50	75.35	30.1	—	—
35	28.99	23.56	7.2	—	—	86	80.71	—	30.8	36	34
36	29.86	—	7.5	—	—	87	81.94	—	31.5	37	35
37	30.74	25.50	7.7	—	—	88	83.19	—	32.2	—	—
38	31.62	—	8.0	—	17	89	84.46	—	33.0	38	36
39	32.50	—	8.3	17	—	90	85.75	81.86	33.8	—	—
40	33.39	27.95	8.6	—	—	91	87.05	—	34.7	39	37
41	34.28	—	8.9	—	—	92	88.37	—	35.5	40	38
42	35.18	—	9.2	—	18	93	89.71	—	36.4	41	—
43	36.08	—	9.5	18	—	94	91.07	—	37.3	—	39
44	36.99	—	9.8	—	—	95	92.46	89.34	38.2	42	40
45	37.90	28.30	10.2	—	—	96	93.89	—	39.2	43	—
46	38.82	—	10.5	19	19	97	95.34	—	40.3	44	41
47	39.74	—	10.9	—	—	98	96.84	—	41.5	45	42
48	40.61	—	11.2	—	—	99	98.39	—	42.7	46	43
49	41.59	—	11.6	—	—	100	100.00	100.00	43.9	47	—
50	42.52	36.46	11.9	20	20						

*Determination of the True Strengths of Spirit for the Standard Temperature of 59° F.*—When for the determination of the strength of a spirit of wine, the indications of the alcoholometer and of the thermometer have been read off, we possess two figures by means of which the true strength of spirit of the spirits of wine to be examined, *i. e.*, the number of liters of absolute alcohol contained in 100 liters of the fluid to be examined, when the latter possesses the standard temperature of 59° F., is found as follows: If the observed temperature of the fluid is = 59° F., which is indicated on the scale of the thermometer with a red mark, the figure read off on the scale of the alcoholometer indicates at once the “true” strength of spirit. If, however, the thermometer shows a different temperature, in which case the figure read off on the scale of the alcoholometer is termed the “apparent” strength of spirit, the true strength of spirit is found from the figure read off on the scale of the alcoholometer and the temperature with the assistance of the following table:



TABLE VI.—*Determination of the true strength of spirits for the Standard temperature of 59° F. (15° C.).*

Temperature, degrees C.	Temperature, degrees F.	31	32	33	34	35	36	37	38	39	40	41
True strength of spirit for the above apparent strengths.												
—25	—13	47.9	48.7	49.5	50.3	51.1	51.9	52.7	53.6	54.4	55.2	56.0
—23.75	—10.75	47.4	48.2	49.0	49.8	50.6	51.5	52.3	53.1	53.9	54.7	55.5
—22.5	—8.5	46.9	47.7	48.5	49.3	50.1	51.0	51.8	52.6	53.4	54.3	55.1
—21.25	—6.25	46.4	47.2	48.0	48.8	49.6	50.5	51.3	52.1	53.0	53.8	54.6
—20	—4	45.8	46.7	47.5	48.3	49.2	50.0	50.8	51.7	52.5	53.3	54.2
—18.75	—1.75	45.3	46.1	47.0	47.8	48.7	49.5	50.3	51.2	52.0	52.9	53.7
—17.5	+0.5	44.8	45.6	46.5	47.3	48.2	49.0	49.9	50.7	51.6	52.4	53.3
—16.25	+2.75	44.2	45.1	46.0	46.8	47.7	48.5	49.4	50.2	51.1	51.9	52.8
—15	+5	43.7	44.6	45.4	46.3	47.2	48.0	48.9	49.8	50.6	51.5	52.3
—13.75	+7.25	43.2	44.1	44.9	45.8	46.7	47.5	48.4	49.3	50.1	51.0	51.9
—12.5	+9.5	42.7	43.5	44.4	45.3	46.2	47.1	47.9	48.8	49.7	50.6	51.4
—11.25	+11.75	42.1	43.0	43.9	44.8	45.7	46.6	47.5	48.3	49.2	50.1	51.0
—10	+14	41.6	42.5	43.4	44.3	45.2	46.1	47.0	47.9	48.8	49.7	50.6
—8.75	+16.25	41.1	42.0	42.9	43.8	44.7	45.6	46.5	47.4	48.3	49.2	50.1
—7.5	+18.5	40.6	41.5	42.4	43.3	44.2	45.1	46.0	46.9	47.9	48.8	49.7
—6	+20.75	40.1	41.0	41.9	42.8	43.7	44.6	45.6	46.5	47.4	48.3	49.2
—5	+23	39.5	40.5	41.4	42.3	43.2	44.2	45.1	46.0	46.9	47.8	48.8
—3.75	+25.25	39.0	39.9	40.9	41.8	42.7	43.7	44.6	45.5	46.4	47.4	48.3
—2.5	+27.5	38.4	39.4	40.3	41.3	42.2	43.2	44.1	45.0	45.9	46.9	47.8
—1.25	+29.75	37.9	38.9	39.8	40.8	41.7	42.7	43.6	44.5	45.5	46.4	47.3
0	+32	37.4	38.3	39.3	40.3	41.2	42.2	43.1	44.0	45.0	45.9	46.9
+1.25	+34.25	36.8	37.8	38.8	39.7	40.7	41.7	42.6	43.5	44.5	45.4	46.4
+2.5	+36.5	36.3	37.3	38.2	39.2	40.2	41.1	42.1	43.0	44.0	45.0	45.9
+3.75	+38.75	35.7	36.7	37.7	38.7	39.7	40.6	41.6	42.5	43.5	44.5	45.4
+5	+41	35.2	36.2	37.2	38.2	39.1	40.1	41.1	42.0	43.0	44.0	44.9
+6.25	+43	34.7	35.7	36.7	37.6	38.6	39.6	40.6	41.5	42.5	43.5	44.5
+7.5	+45.5	34.1	35.1	36.1	37.1	38.1	39.1	40.1	41.0	42.0	43.0	44.0
+8.75	+47.75	33.6	34.6	35.6	36.6	37.6	38.6	39.6	40.5	41.5	42.5	43.5
+10	+50	33.1	34.1	35.1	36.1	37.1	38.1	39.0	40.0	41.0	42.0	43.0
+11.25	+52.25	32.5	33.6	34.6	35.6	36.6	37.5	38.5	39.5	40.5	41.5	42.5
+12.5	+54.5	32.0	33.0	34.0	35.0	36.0	37.0	38.0	39.0	40.0	41.0	42.0
+13.75	+56.75	31.5	32.5	33.5	34.5	35.5	36.5	37.5	38.5	39.5	40.5	41.5
+15	+59	31.0	32.0	33.0	34.0	35.0	36.0	37.0	38.0	39.0	40.0	41.0
+16.25	+61.25	30.5	31.5	32.5	33.5	34.5	35.5	36.5	37.5	38.5	39.5	40.5
+17.5	+63.5	30.0	31.0	32.0	33.0	34.0	35.0	36.0	37.0	38.0	39.0	40.0
+18.75	+65.75	29.5	30.5	31.5	32.5	33.5	34.5	35.5	36.5	37.5	38.5	39.5
+20	+68	29.0	30.0	31.0	31.9	32.9	33.9	35.0	36.0	37.0	38.0	39.0
+21.25	+70.25	28.5	29.5	30.4	31.4	32.4	33.4	34.4	35.5	36.5	37.5	38.5
+22.5	+72.5	28.0	29.0	29.9	30.9	31.9	32.9	33.9	34.9	36.0	37.0	38.0
+23.75	+74.75	27.5	28.5	29.4	30.4	31.4	32.4	33.4	34.5	35.5	36.5	37.5
+25	+77	27.0	28.0	28.9	29.9	30.9	31.9	32.9	33.9	34.9	36.0	37.0
+26.25	+79.25	29.5	27.5	28.4	29.4	30.4	31.4	32.4	33.4	34.4	35.5	36.5
+27.5	+81.5	26.0	27.0	28.0	28.9	29.9	30.9	31.9	32.9	33.9	35.0	36.0
+28.75	+83.75	25.6	26.5	27.5	28.4	29.4	30.4	31.4	32.4	33.4	34.4	35.5
+30	+86	25.1	26.0	27.0	27.9	28.9	29.9	30.9	31.9	32.9	33.9	35.0
+31.25	+88.25	24.6	25.5	26.5	27.4	28.4	29.4	30.4	31.4	32.4	33.4	34.5
+32.5	+90.5	24.1	25.0	26.0	26.9	27.9	28.9	29.9	30.9	31.9	32.9	34.0
+33.75	+92.75	23.6	24.5	25.5	26.4	27.4	28.4	29.4	30.4	31.4	32.4	33.5
+35	+95	23.1	24.1	25.0	25.9	26.9	27.9	28.9	29.9	30.9	31.9	32.9
+36.25	+97.25	22.7	23.6	24.5	25.4	26.4	27.4	28.4	29.4	30.4	31.4	32.4
+37.5	+99.5	22.2	23.1	24.0	24.9	25.9	26.9	27.9	28.9	29.9	30.9	31.9

TABLE VI.—*Continued.*

Temperature, degrees C.	Temperature, degrees F.	42	43	44	45	46	47	48	49	50	51	52
True strength of spirit for the above apparent strengths.												
—25.	—13	56.8	57.6	58.4	59.3	60.1	61.0	61.8	62.7	63.6	64.5	65.4
—23.75	—10.75	56.3	57.2	58.0	58.8	59.7	60.6	61.4	62.3	63.2	64.1	65.0
—22.5	—8.5	55.9	56.7	57.6	58.4	59.3	60.1	61.0	61.9	62.8	63.7	64.6
—21.25	—6.25	55.4	56.3	57.1	58.0	58.8	59.7	60.6	61.5	62.4	63.3	64.2
—20	—4	55.0	55.8	56.7	57.6	58.4	59.3	60.2	61.1	61.9	62.9	63.8
—18.75	—1.75	54.6	55.4	56.3	57.1	58.0	58.9	59.7	60.6	61.5	62.5	63.4
—17.5	+0.5	54.1	55.0	55.8	56.7	57.6	58.4	59.3	60.2	61.1	62.1	63.0
—16.25	+2.25	53.7	54.5	55.4	56.3	57.1	58.0	58.9	59.8	60.7	61.7	62.6
—15	+5	53.2	54.1	55.0	55.8	56.7	57.6	58.5	59.4	60.3	61.2	62.2
—13.75	+7.25	52.8	53.6	54.5	55.4	56.3	57.2	58.1	59.0	59.9	60.8	61.8
—12.5	+9.5	52.3	53.2	54.1	55.0	55.9	56.8	57.7	58.6	59.5	60.4	61.4
—11.25	+11.75	51.9	52.8	53.7	54.5	55.4	56.3	57.2	58.2	59.1	60.0	61.0
—10	+14	51.4	52.3	53.2	54.1	55.0	55.9	56.8	57.8	58.7	59.6	60.6
—8.75	+16.25	51.0	51.9	52.8	53.7	54.6	55.5	56.4	57.3	58.3	59.2	60.2
—7.5	+18.5	50.6	51.5	52.4	53.3	54.2	55.1	56.0	56.9	57.9	58.8	59.8
—6	+20.75	50.1	51.0	51.9	52.9	53.8	54.7	55.6	56.5	57.5	58.4	59.3
—5	+23	49.7	50.6	51.5	52.4	53.3	54.3	55.2	56.1	57.0	58.0	58.9
—3.75	+25.25	49.2	50.1	51.1	52.0	52.9	53.8	54.8	55.7	56.6	57.6	58.5
—2.5	+27.5	48.8	49.7	50.6	51.6	52.5	53.4	54.3	55.3	56.2	57.2	58.1
—1.25	+29.75	48.3	49.2	50.2	51.1	52.0	53.0	53.9	54.9	55.8	56.8	57.7
0	+32	47.8	48.8	49.7	50.7	51.6	52.5	53.5	54.4	55.4	56.3	57.3
+1.25	+34.25	47.4	48.3	49.3	50.2	51.2	52.1	53.0	54.0	54.9	55.9	56.9
+2.5	+36.5	46.9	47.8	48.8	49.8	50.7	51.6	52.6	53.5	54.5	55.5	56.4
+3.75	+38.75	46.4	47.4	48.3	49.3	50.2	51.2	52.1	53.1	54.1	55.0	56.0
+5	+41	45.9	46.9	47.9	48.8	49.8	50.7	51.7	52.7	53.6	54.6	55.6
+6.25	+43	45.4	46.4	47.4	48.3	49.3	50.3	51.2	52.2	53.2	54.2	55.1
+7.5	+45.5	44.9	45.9	46.9	47.9	48.8	49.8	50.8	51.8	52.7	53.7	54.7
+8.75	+47.75	44.5	45.4	46.4	47.4	48.4	49.4	50.3	51.3	52.3	53.3	54.2
+10	+50	44.0	44.9	45.9	46.9	47.9	48.9	49.9	50.9	51.8	52.8	53.8
+11.25	+52.25	43.5	44.5	45.5	46.4	47.4	48.4	49.4	50.4	51.4	52.4	53.4
+12.5	+54.5	43.0	44.0	45.0	46.0	47.0	48.0	48.9	49.9	50.9	51.9	52.9
+13.75	+56.75	42.5	43.5	44.5	45.5	46.5	47.5	48.5	49.5	50.5	51.5	52.5
+15	+59	42.0	43.0	44.0	45.0	46.0	47.0	48.0	49.0	50.0	51.0	52.0
+16.25	+61.25	41.5	42.5	43.5	44.5	45.5	46.4	47.5	48.5	49.5	50.5	51.5
+17.5	+63.5	41.0	42.0	43.0	44.0	45.0	46.0	47.1	48.1	49.1	50.1	51.1
+18.75	+65.75	40.5	41.5	42.5	43.5	44.5	45.6	46.6	47.6	48.6	49.6	50.6
+20	+68	40.0	41.0	42.0	43.1	44.1	45.1	46.1	47.1	48.1	49.1	50.2
+21.25	+70.25	39.5	40.5	41.6	42.6	43.6	44.6	45.6	46.6	47.6	48.7	49.7
+22.5	+72.5	39.0	40.0	41.1	42.1	43.1	44.1	45.1	46.1	47.2	48.2	49.2
+23.75	+74.75	38.5	39.5	40.6	41.6	42.6	43.6	44.6	45.7	46.7	47.7	48.7
+25	+77	38.0	39.0	40.1	41.1	42.1	43.1	44.2	45.2	46.2	47.2	48.3
+26.25	+79.25	37.5	38.5	39.6	40.6	41.6	42.6	43.7	44.7	45.7	46.8	47.8
+27.5	+81.5	37.0	38.0	39.1	40.1	41.1	42.2	43.2	44.2	45.2	46.3	47.3
+28.75	+83.75	36.5	37.5	38.6	39.6	40.6	41.7	42.7	43.7	44.8	45.8	46.8
+30	+86	36.0	37.0	38.1	39.1	40.1	41.2	42.2	43.2	44.3	45.3	46.4
+31.25	+88.25	35.5	36.5	37.6	38.6	39.6	40.7	41.7	42.7	43.8	44.8	45.9
+32.5	+90.5	35.0	36.0	37.1	38.1	39.1	40.2	41.2	42.3	43.3	44.4	45.4
+33.75	+92.75	34.5	35.5	36.6	37.6	38.6	39.7	40.7	41.8	42.8	43.9	44.9
+35	+95	34.0	35.0	36.1	37.1	38.1	39.2	40.2	41.3	42.3	43.4	44.4
+36.25	+97.25	33.5	34.5	35.6	36.6	37.6	38.7	39.7	40.8	41.8	42.9	43.9
+37.5	+99.5	33.0	34.0	35.1	36.1	37.1	38.2	39.2	40.3	41.3	42.4	43.4



TABLE VI.—*Continued.*

Temperature, degrees C.	Temperature, degrees F.	53	54	55	56	57	58	59	60	61	62	63
True strengths of spirit for the above apparent strengths.												
—25	—13	66.3	67.2	68.1	69.1	70.0	70.9	71.8	72.7	73.6	74.4	75.3
—23.75	—10.75	65.9	66.8	67.7	68.7	69.6	70.5	71.4	72.3	73.2	74.1	75.0
—22.5	—8.5	65.5	66.4	67.3	68.3	69.2	70.1	71.0	71.9	72.8	73.7	74.6
—21.25	—6.25	65.1	66.0	67.0	67.9	68.8	69.7	70.6	71.5	72.4	73.3	74.2
—20	—4	64.7	65.6	66.6	67.5	68.4	69.3	70.2	71.1	72.0	72.9	73.9
—18.75	—1.75	64.3	65.2	66.2	67.1	68.0	68.9	69.8	70.8	71.7	72.6	73.5
—17.5	+0.5	63.9	64.8	65.8	66.7	67.6	68.5	69.5	70.4	71.3	72.2	73.1
—16.25	+2.75	63.5	64.4	65.4	66.3	67.2	68.1	69.1	70.0	70.9	71.8	72.7
—15	+5	63.1	64.0	65.0	65.9	66.8	67.8	68.7	69.6	70.5	71.4	72.4
—13.75	+7.25	62.7	63.6	64.6	65.5	66.4	67.4	68.3	69.2	70.2	71.1	72.0
—12.5	+9.5	62.3	63.2	64.2	65.1	66.0	67.0	67.9	68.9	69.8	70.7	71.6
—11.25	+11.75	61.9	62.8	63.8	64.7	65.7	66.6	67.6	68.5	69.4	70.3	71.3
—10	+14	61.5	62.4	63.4	64.3	65.3	66.2	67.2	68.1	69.0	70.0	70.9
—8.75	+16.25	61.1	62.0	63.0	63.9	64.9	65.9	66.8	67.7	68.7	69.6	70.5
—7.5	+18.5	60.7	61.6	62.6	63.6	64.5	65.5	66.4	67.4	68.3	69.2	70.2
—6	+20.75	60.3	61.2	62.2	63.2	64.1	65.1	66.0	67.0	67.9	68.9	69.8
—5	+23	59.9	60.8	61.8	62.8	63.7	64.7	65.6	66.6	67.5	68.5	69.4
—3.75	+25.25	59.5	60.4	61.4	62.4	63.3	64.3	65.3	66.2	67.2	68.1	69.1
—2.5	+27.5	59.1	60.0	61.0	62.0	62.9	63.9	64.9	65.8	66.8	67.7	68.7
—1.25	+29.75	58.7	59.6	60.6	61.6	62.5	63.5	64.5	65.4	66.4	67.3	68.3
0	+32	58.3	59.2	60.2	61.2	62.1	63.1	64.1	65.0	66.0	67.0	67.9
+1.25	+34.25	57.8	58.8	59.8	60.7	61.7	62.7	63.7	64.6	65.6	66.6	67.5
+2.5	+36.5	57.4	58.4	59.3	60.3	61.3	62.3	63.3	64.2	65.2	66.2	67.1
+3.75	+38.75	57.0	57.9	58.9	59.9	60.9	61.9	62.8	63.8	64.8	65.8	66.7
+5	+41	56.5	57.5	58.5	59.5	60.5	61.4	62.4	63.4	64.4	65.4	66.3
+6.25	+43	56.1	57.1	58.1	59.0	60.0	61.0	62.0	63.0	64.0	64.9	65.9
+7.5	+45.5	55.7	56.6	57.6	58.6	59.6	60.6	61.6	62.6	63.5	64.5	65.5
+8.75	+47.75	55.2	56.2	57.2	58.2	59.2	60.2	61.2	62.1	63.1	64.1	65.1
+10	+50	54.8	55.8	56.8	57.8	58.7	59.7	60.7	61.7	62.7	63.7	64.7
+11.25	+52.25	54.3	55.3	56.3	57.3	58.3	59.3	60.3	61.3	62.3	63.3	64.3
+12.5	+54.5	53.9	54.9	55.9	56.9	57.9	58.9	59.9	60.9	61.9	62.9	63.9
+13.75	+56.75	53.5	54.4	55.4	56.4	57.4	58.4	59.4	60.4	61.4	62.4	63.4
+15	+59	53.0	54.0	55.0	56.0	57.0	58.0	59.0	60.0	61.0	62.0	63.0
+16.25	+61.25	52.5	53.5	54.6	55.6	56.6	57.6	58.6	59.6	60.6	61.6	62.6
+17.5	+63.5	52.1	53.1	54.1	55.1	56.1	57.1	58.1	59.1	60.1	61.1	62.1
+18.75	+65.75	51.6	52.6	53.7	54.7	55.7	56.7	57.7	58.7	59.7	60.7	61.7
+20	+68	51.2	52.2	53.2	54.2	55.2	56.2	57.2	58.3	59.3	60.3	61.3
+21.25	+70.25	50.7	51.7	52.7	53.8	54.8	55.8	56.8	57.8	58.8	59.8	60.8
+22.5	+72.5	50.2	51.3	52.3	53.3	54.3	55.3	56.3	57.4	58.4	59.4	60.5
+23.75	+74.75	49.8	50.8	51.8	52.8	53.9	54.9	55.9	56.9	57.9	58.9	60.0
+25	+77	49.3	50.3	51.4	52.4	53.4	54.4	55.5	56.5	57.5	58.5	59.5
+26.25	+79.25	48.8	49.9	50.9	51.9	53.0	54.0	55.0	56.0	57.0	58.1	59.1
+27.5	+81.5	48.4	49.4	50.4	51.5	52.5	53.5	54.5	55.6	56.6	57.6	58.6
+28.75	+83.75	47.9	48.9	50.0	51.0	52.0	53.1	54.1	55.1	56.1	57.2	58.2
+30	+86	47.4	48.4	49.5	50.5	51.6	52.6	53.6	54.7	55.7	56.7	57.7
+31.25	+88.25	46.9	48.0	49.0	50.1	51.1	52.1	53.2	54.2	55.2	56.2	57.3
+32.5	+90.5	46.4	47.5	48.5	49.6	50.6	51.7	52.7	53.7	54.8	55.8	56.8
+33.75	+92.75	46.0	47.0	48.1	49.1	50.2	51.2	52.2	53.3	54.3	55.3	56.4
+35	+95	45.5	46.5	47.6	48.6	49.7	50.7	51.8	52.8	53.8	54.9	55.9
+36.25	+97.25	45.0	46.0	47.1	48.2	49.2	50.3	51.3	52.4	53.4	54.4	55.4
+37.5	+99.5	44.5	45.6	46.6	47.7	48.7	49.8	50.8	51.9	52.9	53.9	55.0



TABLE VI.—*Continued.*

Temperature, degrees C.	Temperature, degrees F.	64	65	66	67	68	69	70	71	72	73	74
True strength of spirit for the above apparent strengths.												
—25	—13	76.2	77.1	78.0	79.0	79.9	80.8	81.7	82.6	83.5	84.4	85.3
—23.75	—10.75	75.9	76.8	77.7	78.6	79.5	80.4	81.3	82.2	83.2	84.1	85.0
—22.5	—8.5	75.5	76.4	77.3	78.2	79.1	80.1	81.0	81.9	82.8	83.7	84.6
—21.25	—6.25	75.1	76.0	77.0	77.9	78.8	79.7	80.6	81.6	82.5	83.4	84.3
—20	—4	74.8	75.7	76.6	77.5	78.4	79.3	80.3	81.2	82.1	83.0	84.0
—18.75	—1.75	74.4	75.3	76.2	77.2	78.1	79.0	79.9	80.9	81.8	82.7	83.6
—17.5	+0.5	74.0	74.9	75.9	76.8	77.7	78.6	79.6	80.5	81.4	82.4	83.3
—16.25	+2.75	73.7	74.6	75.4	76.4	77.4	78.3	79.2	80.2	81.1	82.0	82.9
—15	+5	73.3	74.2	75.1	76.1	77.0	77.9	78.9	79.8	80.7	81.7	82.6
—13.75	+7.25	72.9	73.9	74.8	75.7	76.7	77.6	78.5	79.5	80.4	81.3	82.3
—12.5	+9.5	72.6	73.5	74.4	75.4	76.3	77.2	78.2	79.1	80.1	81.0	81.9
—11.25	+11.75	72.2	73.1	74.1	75.0	75.9	76.9	77.8	78.8	79.7	80.7	81.6
—10	+14	71.8	72.8	73.7	74.6	75.6	76.5	77.5	78.4	79.4	80.3	81.3
—8.75	+16.25	71.5	72.4	73.4	74.3	75.2	76.2	77.1	78.1	79.0	80.0	80.9
—7.5	+18.5	71.1	72.1	73.0	73.9	74.9	75.8	76.8	77.7	78.7	79.6	80.6
—6	+20.75	70.7	71.7	72.6	73.6	74.5	75.5	76.4	77.4	78.4	79.3	80.3
—5	+23	70.4	71.3	72.3	73.2	74.2	75.1	76.1	77.0	78.0	79.0	79.9
—3.75	+25.25	70.0	71.0	71.9	72.9	73.8	74.8	75.7	76.7	77.7	78.6	79.6
—2.5	+27.5	69.6	70.6	71.5	72.5	73.5	74.4	75.4	76.3	77.3	78.3	79.2
—1.25	+29.75	69.3	70.2	71.2	72.1	73.1	74.0	75.0	76.0	77.0	77.9	78.9
0	+32	68.9	69.8	70.8	71.8	72.7	73.7	74.7	75.6	76.7	77.6	78.5
+1.25	+34.25	68.5	69.5	70.4	71.4	72.3	73.3	74.3	75.3	76.2	77.2	78.2
+2.5	+36.5	68.1	69.1	70.0	71.0	72.0	72.9	73.9	74.9	75.9	76.8	77.8
+3.75	+38.75	67.7	68.7	69.6	70.6	71.6	72.6	73.5	74.5	75.5	76.5	77.4
+5	+41	67.3	68.3	69.9	70.2	71.2	72.2	73.1	74.1	75.1	76.1	77.1
+6.25	+43	66.9	67.9	69.3	69.8	70.8	71.8	72.8	73.7	74.7	75.7	76.7
+7.5	+45.5	66.5	67.5	68.5	69.4	70.4	71.4	72.4	73.4	74.3	75.3	76.3
+8.75	+47.75	66.1	67.1	68.1	69.0	70.0	71.0	72.0	73.0	74.0	74.9	75.9
+10	+50	65.7	66.7	67.6	68.6	69.6	70.6	71.6	72.6	73.6	74.6	75.5
+11.25	+52.25	65.3	66.3	67.2	68.2	69.2	70.2	71.2	72.2	73.2	74.2	75.2
+12.5	+54.5	64.8	65.8	66.8	67.8	68.8	69.8	70.8	71.8	72.8	73.8	74.8
+13.75	+56.75	64.4	65.4	66.4	67.4	68.4	69.4	70.4	71.4	72.4	73.4	74.4
+15	+59	64.0	65.0	66.0	67.0	68.0	69.0	70.0	71.0	72.0	73.0	74.0
+16.25	+61.25	63.6	64.6	65.6	66.6	67.6	68.6	69.6	70.6	71.6	72.6	73.6
+17.5	+63.5	63.2	64.2	65.2	66.2	67.2	68.2	69.2	70.2	71.2	72.2	73.2
+18.75	+65.75	62.7	63.7	64.7	65.8	66.8	67.8	68.8	69.8	70.8	71.8	72.8
+20	+68	62.3	63.3	64.3	65.3	66.3	67.4	68.4	69.4	70.4	71.4	72.4
+21.25	+70.25	61.9	62.9	63.9	64.9	65.9	66.9	68.0	69.0	70.0	71.0	72.0
+22.5	+72.5	61.4	62.4	63.5	64.5	65.6	66.5	67.5	68.6	69.6	70.6	71.6
+23.75	+74.75	61.0	62.0	63.0	64.1	65.1	66.1	67.1	68.1	69.2	70.2	71.2
+25	+77	60.5	61.6	62.6	63.6	64.6	65.7	66.7	67.7	68.7	69.8	70.8
+26.25	+79.25	60.1	61.1	62.1	63.2	64.2	65.2	66.3	67.3	68.3	69.4	70.4
+27.5	+81.5	59.6	60.7	61.7	62.7	63.8	64.8	65.8	66.9	67.9	68.9	70.0
+28.75	+83.75	59.2	60.2	61.3	62.3	63.3	64.4	65.4	66.4	67.5	68.5	69.5
+30	+86	58.7	59.8	60.8	61.9	62.9	63.9	65.0	66.0	67.1	68.1	69.1
+31.25	+88.25	58.3	59.3	60.4	61.4	62.5	63.5	64.5	65.6	66.6	67.7	68.7
+32.5	+90.5	57.8	58.9	59.9	61.0	62.0	63.1	64.1	65.1	66.2	67.2	68.3
+33.75	+92.75	57.4	58.4	59.5	60.5	61.6	62.6	63.7	64.7	65.8	66.8	67.9
+35	+95	56.9	58.0	59.0	60.1	61.1	62.2	63.2	64.3	65.3	66.4	67.4
+36.25	+97.25	56.5	57.5	58.6	59.6	60.7	61.7	62.8	63.8	64.9	65.9	67.0
+37.5	+99.5	56.0	57.1	58.1	59.2	60.2	61.3	62.3	63.4	64.4	65.5	66.6

TABLE VI.—*Concluded.*

Temperature, degrees C.	Temperature, degrees F.	75	76	77	78	79	80	81	82	83	84	85	86
True strength of spirit for the above apparent strengths.													
—25	—13	86.2	87.1	88.0	88.9	89.7	90.6	91.1	92.3	93.1	93.9	94.7	95.5
—23.75	—10.75	85.9	86.8	87.7	88.5	89.4	90.3	91.4	92.0	92.8	93.6	94.4	95.3
—22.5	—8.5	85.5	86.4	87.3	88.2	89.1	90.0	90.8	91.7	92.5	93.3	94.2	95.0
—21.25	—6.25	85.2	86.1	87.0	87.9	88.8	89.6	90.5	91.4	92.2	93.0	93.9	94.7
—20	—4	84.9	85.8	86.7	87.6	88.5	89.3	90.2	91.1	91.9	92.8	93.6	94.4
—18.75	—1.75	84.5	85.4	86.3	87.2	88.1	89.0	89.9	90.8	91.6	92.5	93.3	94.1
—17.5	+0.5	84.2	85.1	86.0	86.9	87.8	88.7	89.6	90.4	91.3	92.2	93.0	93.8
—16.25	+2.75	83.9	84.8	85.7	86.6	87.5	88.4	89.3	90.1	91.0	91.9	92.7	93.6
—15	+5	83.5	84.4	85.4	86.3	87.2	88.1	88.9	89.8	90.7	91.6	92.4	93.3
—13.75	+7.25	83.2	84.1	85.0	85.9	86.8	87.7	88.6	89.5	90.4	91.3	92.1	93.0
—12.5	+9.5	82.9	83.8	84.7	85.6	86.5	87.4	88.3	89.2	90.1	91.0	91.9	92.7
—11.25	+11.75	82.5	83.5	84.4	85.3	86.2	87.1	88.0	88.9	89.8	90.7	91.6	92.5
—10	+14	82.2	83.1	84.1	85.0	85.9	86.8	87.7	88.6	89.5	90.4	91.3	92.2
—8.75	+16.25	81.9	82.8	83.7	84.7	85.6	86.5	87.4	88.3	89.2	90.1	91.0	91.9
—7.5	+18.5	81.5	82.5	83.4	84.3	85.3	86.2	87.1	88.0	88.9	89.8	90.7	91.6
—6	+20.75	81.2	82.1	83.1	84.0	85.0	85.9	86.8	87.7	88.6	89.5	90.5	91.4
—5	+23	80.9	81.9	82.8	83.7	84.6	85.6	86.5	87.4	88.3	89.2	90.2	91.1
—3.75	+25.25	80.5	81.5	82.4	83.4	84.3	85.2	86.2	87.1	88.0	88.9	89.9	90.8
—2.5	+27.5	80.2	81.1	82.1	83.0	84.0	84.9	85.9	86.8	87.7	88.6	89.6	90.5
—1.25	+29.75	79.8	80.8	81.8	82.7	83.7	84.6	85.5	86.5	87.4	88.3	89.3	90.2
0	+32	79.5	80.4	81.4	82.4	83.3	84.3	85.2	86.2	87.1	88.0	89.0	89.9
+1.25	+34.25	79.1	80.1	81.1	82.0	83.0	83.9	84.9	85.8	86.8	87.7	88.6	89.6
+2.5	+36.5	78.8	79.7	80.7	81.7	82.6	83.6	84.5	85.5	86.4	87.4	88.3	89.3
+3.75	+38.75	78.4	79.4	80.3	81.3	82.3	83.2	84.2	85.2	86.1	87.1	88.0	89.0
+5	+41	78.0	79.0	80.0	81.0	81.9	82.9	83.9	84.8	85.8	86.7	87.7	88.6
+6.25	+43	77.7	78.6	79.6	80.6	81.6	82.5	83.5	84.5	85.4	86.4	87.4	88.3
+7.5	+45.5	77.3	78.3	79.3	80.2	81.2	82.2	83.2	84.1	85.1	86.1	87.0	88.0
+8.75	+47.75	76.9	77.9	78.9	79.9	80.8	81.8	82.8	83.8	84.8	85.7	86.7	87.7
+10	+50	76.5	77.5	78.5	79.5	80.5	81.5	82.4	83.4	84.4	85.4	86.4	87.3
+11.25	+52.25	76.2	77.1	78.1	79.1	80.1	81.1	82.1	83.1	84.1	85.0	86.0	87.0
+12.5	+54.5	75.8	76.8	77.8	78.8	79.7	80.7	81.7	82.7	83.7	84.7	85.7	86.7
+13.75	+56.75	75.4	76.4	77.4	78.4	79.4	80.4	81.4	82.4	83.4	84.3	85.3	86.3
+15	+59	75.0	76.0	77.0	78.0	79.0	80.0	81.0	82.0	83.0	84.0	85.0	86.0
+16.25	+61.25	74.6	75.6	76.6	77.6	78.6	79.6	80.6	81.6	82.6	83.6	84.6	85.6
+17.5	+63.5	74.2	75.2	76.2	77.2	78.2	79.2	80.3	81.3	82.3	83.3	84.3	85.3
+18.75	+65.75	73.8	74.8	75.8	76.8	77.9	78.7	79.9	80.9	81.9	82.9	83.9	84.9
+20	+68	73.4	74.4	75.1	76.5	77.5	78.5	79.5	80.5	81.5	82.6	83.6	84.6
+21.25	+70.25	73.0	74.0	74.7	76.1	77.1	78.1	79.1	80.1	81.2	82.2	83.2	84.2
+22.5	+72.5	72.6	73.6	74.4	75.7	76.7	77.7	78.7	79.8	80.8	81.8	82.9	83.9
+23.75	+74.75	72.2	73.2	74.3	75.3	76.3	77.3	78.4	79.4	80.4	81.5	82.5	83.5
+25	+77	71.8	72.8	73.9	74.9	75.9	76.9	78.0	79.0	80.0	81.1	82.1	83.2
+26.25	+79.25	71.4	72.4	73.5	74.5	75.5	76.5	77.6	78.6	79.6	80.7	81.7	82.8
+27.5	+81.5	71.0	72.0	73.0	74.1	75.1	76.1	77.2	78.2	79.3	80.3	81.4	82.4
+28.75	+83.75	70.6	71.6	72.6	73.7	74.7	75.7	76.8	77.8	78.9	79.9	81.0	82.0
+30	+86	70.2	71.2	72.2	73.3	74.3	75.3	76.4	77.4	78.5	79.5	80.6	81.7
+31.25	+88.25	69.7	70.8	71.8	72.9	73.9	74.9	76.0	77.0	78.1	79.1	80.2	81.3
+32.5	+90.5	69.3	70.4	71.4	72.4	73.5	74.5	75.6	76.6	77.7	78.7	79.8	80.9
+33.75	+92.75	68.9	69.9	71.0	72.0	73.1	74.1	75.2	76.2	77.3	78.3	79.4	80.5
+35	+95	68.5	69.5	70.6	71.6	72.7	73.7	74.8	75.8	76.9	77.9	79.0	80.1
+36.25	+97.25	68.1	69.1	70.1	71.2	72.2	73.3	74.3	75.4	76.5	77.5	78.6	79.7
+37.5	+99.5	67.6	68.7	69.7	70.8	71.8	72.9	73.9	75.0	76.1	77.1	78.2	79.3

Table VI. has two entries : one in the uppermost horizontal line for the observed statements of the alcoholometer, hence the apparent strengths from 31 to 44 per cent. ; the other in the first vertical column for the statements of Fahrenheit's thermometer from  $-13^{\circ}$  to  $+99.5^{\circ}$ . On the place where a vertical and horizontal column cross, the strength corresponding to the normal temperature of  $59^{\circ}$  F., *i. e.* the true strength of spirits, is found.

If, for instance, the alcoholometer immersed into a sample of spirits of wine indicates an apparent strength of 77 per cent., and the thermometer the temperature of the fluid as  $25.5^{\circ}$  F., the figure 77 has to be found in the uppermost horizontal column, and then the vertical column belonging to it is followed downward until the horizontal line is reached in which stands the figure 25.5 in the column containing the degrees of temperature. Here the statement 82.4 will be found as the true strength of spirit, and this figure indicates that at the normal temperature of  $59^{\circ}$  F. 100 liters of the spirit of wine examined contain 82.4 liters of absolute alcohol.

When the apparent strength read off on the alcoholometer consists of a whole number and a fraction, the true strength corresponding to the whole number is determined in the above manner, and the surplus fraction added to the number found.

If, for instance, the temperature read off is  $74.75^{\circ}$ , and the apparent strength  $81\frac{3}{4}$  per cent., the true strength belonging to 81 per cent. and  $74.75^{\circ}$ , which is  $= 78.4$ , is first found in the table, and to this is added the fraction  $\frac{3}{4} = 0.72$ , or sufficiently accurate  $= 0.7$ . This gives  $78.4 + 0.7 = 79.1$  per cent. as the nearest accurate true strength.



TABLE VII.—*Determination of the true volume of alcoholic fluids from the apparent volume at different temperatures.*

(According to A. F. W. Brix.)

Degrees C.	Degrees F.	55-57	58-60	61-64	65-69	70-74	75-79	80-84	85-89	90-94
Reducing factors for the above strengths of spirits of wine.										
-10	+14	1.0198	1.0203	1.0207	1.0213	1.0220	1.0227	1.0233	1.0238	1.0246
-8.75	+16.25	0189	0193	0197	0203	0210	0217	0222	0227	0235
-7.5	+18.5	0180	0183	0187	0193	0200	0206	0211	0216	0223
-6	+20.75	0170	0173	0177	0183	0189	0195	0200	0205	0211
-5	+23	0161	0164	0168	0173	0179	0185	0190	0194	0200
-3.75	+25.25	0152	0155	0158	0163	0169	0175	0179	0183	0189
-2.5	+27.5	0143	0146	0148	0153	0159	0164	0168	0172	0178
-1.25	+29.75	0133	0136	0138	0143	0148	0153	0157	0161	0166
0	+32	0123	0126	0128	0132	0138	0142	0146	0150	0154
+1.25	+34.25	0114	0117	0118	0122	0127	0131	0135	0139	0142
+2.5	+36.5	0105	0107	0108	0112	0116	0120	0124	0128	0130
+3.75	+38.75	0095	0097	0098	0102	0105	0109	0113	0116	0118
+5	+41	0085	0087	0088	0091	0094	0098	0101	0104	0106
+6.25	+43.25	0075	0077	0078	0080	0083	0086	0089	0092	0094
+7.5	+45.5	0066	0067	0068	0070	0072	0075	0078	0080	0082
+8.75	+47.75	0056	0057	0058	0060	0061	0064	0066	0068	0070
+10	+50	0046	0047	0048	0050	0050	0053	0054	0056	0058
+11.25	+52.25	0036	0037	0038	0039	0039	0041	0042	0044	0045
+12.5	+54.5	0026	0027	0027	0028	0028	0029	0030	0031	0032
+13.75	+56.75	0015	0016	0016	0017	0017	0017	0018	0019	0019
+15	+59	1.0005	1.0005	1.0005	1.0005	1.0006	1.0006	1.0006	1.0006	1.0006
+16.25	+61.25	0.9995	0.9995	0.9995	0.9995	0.9994	0.9994	0.9994	0.9994	0.9993
+17.5	+63.5	9985	9985	9984	9984	9983	9983	9982	9982	9981
+18.75	+65.75	9975	9975	9974	9973	9972	9971	9970	9969	9968
+20	+68	9965	9965	9963	9962	9961	9960	9958	9957	9955
+21.25	+70.25	9955	9955	9952	9951	9950	9949	9946	9945	9942
+22.5	+72.5	9945	9944	9941	9940	9939	9937	9934	9932	9929
+23.75	+74.75	9934	9933	9930	9929	9927	9925	9922	9919	9916
+25	+77	9923	9922	9919	9917	9915	9912	9909	9906	9903
+26.25	+79.25	9912	9911	9908	9906	9903	9901	9897	9893	9890
+27.5	+81.5	9901	9900	9897	9894	9891	9889	9885	9880	9877
+28.75	+83.75	9890	9889	9886	9882	9879	9876	9872	9867	9864
+30	+86	9879	9877	9874	9870	9866	9863	9859	9854	9851
+31.25	+88.25	0.9868	0.9865	0.9862	0.9858	0.9854	0.9850	0.9846	0.9841	0.9837

*Explanation of Table VII.*

Alcohol, or alcohol and water, heated above or cooled below the standard temperature, expands or contracts. Now, for instance, what is the volume of 10,000 liters of a mixture of 82 per cent. by volume at +5° C. (41° F.) at the standard tem-

perature.\* In the horizontal column below 80–84 and in the vertical column at  $+5^{\circ}$  C. ( $41^{\circ}$  F.) is the reducing factor 1.0101; hence 10,000 liters are  $10,000 \times 1.0101 = 10,101$  liters. 82 being exactly the mean of 80–84, the reduction is accurate. At  $83^{\circ}$  the factor would have to be increased by  $\frac{1}{6}$ ; at 84 by  $\frac{2}{6}$ , and consequently the factor for  $83^{\circ}$  would be  $1.0101 + (1.0104 - 1.0101) \frac{1}{6} = 1.01016$ . For the practice the above factors suffice without change. The measuring of the temperature and reading-off of the percentage of the spirit should be done in the storage-cellar, and not in a warmer room, for instance, the office, as is frequently the custom to the disadvantage of the seller.

\* This table is calculated for a standard temperature of  $15.5^{\circ}$  C. ( $60^{\circ}$  F.).

TABLE VIII.—*Preparation of whiskey of various strengths from spirits of wine.*  
(According to Brix.)

		From 100 liters of a spirit of wine of per cent.															
		94	93	92	91	90	89	88	87	86	85	84	83	82	81	80	79
To prepare whiskey of per cent.																	
		Liters of water must be added.															
30	220.5	216.9	213.4	209.8	206.3	202.7	199.2	195.6	192.1	188.6	185.1	181.6	178.1	174.6	171.1	167.6	164.1
31	210.4	206.9	203.5	200.0	196.6	193.1	189.7	186.3	182.9	179.4	176.0	172.6	169.2	165.8	162.5	159.1	155.7
32	200.9	197.5	194.2	190.8	187.5	184.1	180.8	177.5	174.2	170.9	167.6	164.3	161.0	157.7	154.4	151.1	147.8
33	192.0	188.7	185.4	182.2	178.9	175.7	172.4	169.2	166.0	162.8	159.6	156.4	153.2	150.0	146.8	143.6	140.4
34	183.5	180.4	177.2	174.0	170.9	167.7	164.6	161.4	158.3	155.2	152.0	148.9	145.8	142.7	139.6	136.5	133.4
35	175.6	172.5	169.4	166.3	163.3	160.2	157.1	154.1	151.0	148.0	144.9	141.9	138.9	135.9	132.8	129.7	126.6
36	168.1	165.1	162.1	159.1	156.1	153.1	150.1	147.1	144.2	141.2	138.2	135.3	132.4	129.4	126.5	123.6	120.7
37	161.0	158.0	155.1	152.2	149.3	146.3	143.4	140.5	137.7	134.8	131.9	129.0	126.1	123.3	120.4	117.6	114.7
38	154.2	151.4	148.5	145.6	142.8	139.9	137.1	134.3	131.5	128.7	125.8	123.0	120.3	117.5	114.7	111.9	109.2
39	147.8	145.0	142.2	139.4	136.6	133.9	131.1	128.3	125.6	122.9	120.1	117.4	114.7	111.9	109.2	106.5	103.8
40	141.7	139.0	136.2	133.5	130.8	128.1	125.4	122.7	120.0	117.3	114.6	112.0	109.3	106.7	104.0	101.4	98.7
41	135.9	133.2	130.5	127.9	125.2	122.6	119.9	117.3	114.7	112.2	109.6	107.0	104.5	101.9	99.4	96.8	94.3
42	130.3	127.7	125.1	122.5	119.9	117.3	114.7	112.2	109.6	107.0	104.5	101.9	99.4	96.8	94.3	91.8	89.3
43	125.0	122.4	119.9	117.3	114.8	112.3	109.8	107.2	104.7	102.2	99.7	97.2	94.8	92.3	89.8	87.3	84.8
44	119.9	117.4	114.9	112.4	109.9	107.5	105.0	102.5	100.1	97.6	95.2	92.8	90.3	87.9	85.5	83.1	80.7
45	115.1	112.5	110.2	107.7	105.3	102.9	100.5	98.0	95.7	93.3	90.9	88.5	86.1	83.7	81.3	79.0	76.6
46	110.4	108.8	106.6	104.2	101.9	99.5	97.1	94.7	92.4	90.1	87.7	85.4	83.0	80.7	78.4	76.0	73.7
47	106.0	103.6	101.3	98.9	96.6	94.3	91.9	89.6	87.3	85.0	82.7	80.4	78.2	75.9	73.6	71.3	69.0
48	101.7	99.4	97.1	94.8	92.5	90.2	87.9	85.7	83.4	81.2	79.0	76.7	74.4	72.2	70.0	67.8	65.5
49	97.6	95.3	93.1	90.8	88.6	86.3	84.1	81.9	79.7	77.4	75.2	73.0	70.8	68.6	66.5	64.3	62.1
50	93.6	91.4	89.2	87.0	84.8	82.6	80.4	78.2	76.0	73.9	71.7	69.5	67.4	65.2	63.1	61.0	58.8



TABLE IX.—*For the reduction of specific gravities to saccharometer per cent.*

(According to Balling.)—Temperature 63.5° F.

Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.
1.0000	0.000	1.0040	1.000	1.0080	2.000	1.0120	3.000	1.0160	4.000	1.0200	5.000
1.0001	0.025	41	025	81	025	121	025	161	025	201	025
2	050	42	050	82	050	122	050	162	050	202	050
3	075	43	075	83	075	123	075	163	075	203	075
4	100	44	100	84	100	124	100	164	100	204	100
5	125	45	125	85	125	125	125	165	125	205	125
6	150	46	150	86	150	126	150	166	150	206	150
7	175	47	175	87	175	127	175	167	175	207	175
8	200	48	200	88	200	128	200	168	200	208	200
9	225	49	225	89	225	129	225	169	225	209	225
1.0010	250	1.0050	250	1.0090	250	1.0130	250	1.0170	250	1.0210	250
11	275	51	275	91	275	131	275	171	275	211	275
12	300	52	300	92	300	132	300	172	300	212	300
13	325	53	325	93	325	133	325	173	325	213	325
14	350	54	350	94	350	134	350	174	350	214	350
15	375	55	375	95	375	135	375	175	375	215	375
16	400	56	400	96	400	136	400	176	400	216	400
17	425	57	425	97	425	137	425	177	425	217	425
18	450	58	450	98	450	138	450	178	450	218	450
19	475	59	475	99	475	139	475	179	475	219	475
1.0020	500	1.0060	500	1.0100	500	1.0140	500	1.0180	500	1.0220	500
21	525	61	525	101	525	141	525	181	525	221	525
22	550	62	550	102	550	142	550	182	550	222	550
23	575	63	575	103	575	143	575	183	575	223	575
24	600	64	600	104	600	144	600	184	600	224	600
25	625	65	625	105	625	145	625	185	625	225	625
26	650	66	650	106	650	146	650	186	650	226	650
27	675	67	675	107	675	147	675	187	675	227	675
28	700	68	700	108	700	148	700	188	700	228	700
29	725	69	725	109	725	149	725	189	725	229	725
1.0030	750	1.0070	750	1.0110	750	1.0150	750	1.0190	750	1.0230	750
31	775	71	775	111	775	151	775	191	775	231	775
32	800	72	800	112	800	152	800	192	800	232	800
33	825	73	825	113	825	153	825	193	825	233	825
34	850	74	850	114	850	154	850	194	850	234	850
35	875	75	875	115	875	155	875	195	875	235	875
36	900	76	900	116	900	156	900	196	900	236	900
37	925	77	925	117	925	157	925	197	925	237	925
38	950	78	950	118	950	158	950	198	950	238	950
39	975	79	975	119	975	159	975	199	975	239	975

TABLE IX.—*Continued.*

Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.
1.0240	6.000	1.0290	7.219	1.0340	8.438	1.0390	9.657	1.0440	10.857	1.0490	12.047
241	024	291	244	341	463	391	681	441	881	491	071
242	048	292	268	342	488	392	706	442	904	492	095
243	073	293	292	343	512	393	731	443	928	493	119
244	097	294	316	344	536	394	756	444	952	494	142
245	122	295	341	345	560	395	780	445	976	495	166
246	146	296	365	346	584	396	804	446	1.000	496	190
247	170	297	389	347	609	397	828	447	023	497	214
248	195	298	413	348	633	398	853	448	047	498	238
249	219	299	438	349	657	399	877	449	081	499	261
1.0250	244	1.0300	463	1.0350	681	1.0400	901	1.0450	095	1.0500	285
251	268	301	488	351	706	401	925	451	119	501	309
252	292	302	512	352	731	402	950	452	142	502	333
253	316	303	536	353	756	403	973	453	166	503	357
254	341	304	560	354	780	404	10.000	454	190	504	381
255	365	305	584	355	804	405	023	455	213	505	404
256	389	306	609	356	828	406	047	456	238	506	428
257	413	307	633	357	853	407	071	457	261	507	452
258	438	308	667	358	877	408	095	458	285	508	476
259	463	309	681	359	901	409	119	459	309	509	500
1.0260	488	1.0310	706	1.0360	925	1.0410	142	1.0460	333	1.0510	523
261	512	311	731	361	950	411	166	461	359	511	547
262	536	312	756	362	975	412	190	462	381	512	571
263	560	313	780	363	9.000	413	214	463	404	513	595
264	584	314	804	364	024	414	238	464	428	514	619
265	609	315	828	365	048	415	261	465	452	515	642
266	633	316	853	366	073	416	285	466	476	516	666
267	657	317	877	367	097	417	309	467	500	517	690
268	681	318	901	368	122	418	333	468	523	518	714
269	706	319	925	369	146	419	357	469	547	519	738
1.0270	731	1.0320	950	1.0370	170	1.0420	381	1.0470	571	1.0520	761
271	756	321	975	371	195	421	404	471	595	521	785
272	780	322	8.000	372	219	422	428	472	619	522	809
273	804	323	024	373	244	423	452	473	642	523	833
274	828	324	048	374	268	424	476	474	676	524	857
275	853	325	073	375	292	425	500	475	690	525	881
276	877	326	097	376	316	426	523	476	714	526	904
277	901	327	122	377	341	427	547	477	738	527	928
278	925	328	146	378	365	428	571	478	761	528	952
279	950	329	170	379	389	429	595	479	785	529	976
1.0280	975	1.0330	195	1.0380	413	1.0430	619	1.0480	809	1.0530	13.000
281	7.000	331	219	381	438	431	642	481	833	531	023
282	024	332	244	382	463	432	666	482	857	532	047
283	048	333	268	383	488	433	690	483	881	533	071
284	073	334	292	384	512	434	714	484	904	534	095
285	097	335	316	385	536	435	738	485	928	535	119
286	122	336	341	386	560	436	761	486	952	536	142
287	146	337	365	387	584	437	785	487	976	537	166
288	170	338	389	388	609	438	809	488	12.000	538	190
289	195	339	413	389	633	439	833	489	023	539	214

TABLE IX.—*Concluded.*

Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.	Specific gravity.	Corresponding saccharometer, statement in per cent.
1.0540	13.238	1.0577	14.119	1.0614	15.000	1.0651	15.860	1.0688	16.721	1.0730	17.681
541	261	578	142	615	024	652	883	689	744	732	725
542	285	579	166	616	046	653	907	1.0690	767	734	772
543	309	1.0580	190	617	070	654	930	691	790	736	818
544	333	581	214	618	093	655	953	692	814	738	863
545	357	582	238	619	116	656	976	693	837	1.0740	909
546	381	583	261	1.0620	139	657	16.000	694	860	742	954
547	404	584	285	621	162	658	023	695	883	744	18.000
548	428	585	309	622	186	659	046	696	907	746	045
549	452	586	333	623	209	1.0660	070	697	930	748	090
1.0550	476	587	357	624	232	661	093	698	953	1.0750	137
551	500	588	381	625	255	662	116	699	976	752	181
552	523	589	404	626	278	663	139	1.0700	17.000	754	227
553	547	1.0590	428	627	302	664	162	701	022	756	272
554	571	591	452	628	325	665	186	702	045	758	318
555	595	592	476	629	348	666	209	703	067	1.0760	363
556	619	593	500	1.0630	371	667	232	704	090	762	409
557	642	594	523	631	395	668	255	705	113	764	454
558	666	595	547	632	418	669	278	706	136	766	500
559	690	596	571	633	441	1.0670	302	707	158	768	545
1.0560	714	597	595	634	464	671	325	708	181	1.0770	590
561	736	598	619	635	488	672	348	709	204	772	636
562	761	599	642	636	511	673	371	1.0710	227	774	681
563	785	1.0600	665	637	534	674	395	711	250	776	727
564	809	601	690	638	557	675	418	712	272	778	772
565	833	602	714	639	581	676	441	713	295	1.0780	818
566	857	603	738	1.0640	604	677	464	714	318	782	863
567	881	604	761	641	627	678	480	715	340	784	909
568	904	605	785	642	650	679	511	716	363	786	954
569	928	606	809	643	674	1.0680	534	717	386	788	19.000
1.0570	952	607	833	644	697	681	557	718	409	1.0790	045
571	976	608	857	645	721	682	581	719	431	792	090
572	14.000	609	881	646	744	683	604	1.0720	454	794	136
573	023	1.0610	904	647	767	684	627	722	500	796	181
574	047	611	928	648	790	685	650	724	545	798	227
575	071	612	952	649	814	686	674	726	590	1.0800	19.272
576	095	613	976	1.0650	837	687	697	728	636		



TABLE X.—*Comparative synopsis of the aerometers for must generally used.*

Specific gravity, degrees (Oechsle).	Extract, per cent. by weight (Balling).	Sugar, per cent. by weight.		Degrees (Wagner).	Specific gravity, degrees (Oechsle).	Extract, per cent. by weight (Balling).	Sugar, per cent. by weight.		Degrees (Wagner).
		Babo.	Pillitz.				Babo.	Pillitz.	
1.051	12.5	10.5	8.2	7	1.091	21.8	18.3	17.5	—
52	12.8	10.7	8.5	—	92	22.1	18.5	17.8	—
53	13.0	10.9	8.7	—	93	22.3	18.6	18.0	—
54	13.2	11.1	8.9	—	94	22.5	18.8	18.2	—
55	13.5	11.3	9.1	—	95	22.7	18.9	18.4	—
56	13.7	11.5	9.4	—	96	22.9	19.0	18.6	—
57	14.0	11.7	9.7	—	97	23.1	19.2	18.8	—
58	14.2	12.0	9.9	8	98	23.3	19.3	19.0	—
59	14.4	12.2	10.1	—	99	23.5	19.5	19.2	13
60	14.7	12.4	10.4	—	1.100	23.7	19.7	19.4	—
61	14.9	12.6	10.6	—	01	23.9	19.9	19.6	—
62	15.1	12.8	10.8	—	02	24.2	20.1	19.9	—
63	15.4	13.0	11.1	—	03	24.4	20.3	20.1	—
64	15.6	13.3	11.3	—	04	24.6	20.5	20.3	—
65	15.8	13.5	11.5	—	05	24.8	20.8	20.5	—
66	16.1	13.7	11.8	9	06	25.0	21.0	20.7	—
67	16.3	13.9	12.0	—	07	25.2	21.2	20.9	14
68	16.5	14.1	12.2	—	08	25.4	21.4	21.1	—
69	16.8	14.3	12.5	—	09	25.7	21.6	21.4	—
70	17.0	14.4	12.7	—	10	25.9	21.8	21.6	—
71	17.2	14.6	12.9	—	11	26.1	22.0	21.8	—
72	17.5	14.8	13.2	—	12	26.3	22.2	22.0	—
73	17.7	15.0	13.4	—	13	26.5	22.4	22.2	—
74	17.9	15.2	13.6	10	14	26.7	22.6	22.4	—
75	18.1	15.4	13.8	—	15	26.9	22.8	22.6	—
76	18.4	15.6	14.1	—	16	27.1	23.0	22.8	15
77	18.6	15.8	14.3	—	17	27.4	23.2	23.1	—
78	18.8	15.9	14.5	—	18	27.6	23.5	23.3	—
79	19.0	16.1	14.7	—	19	27.8	23.8	23.5	—
80	19.3	16.3	15.0	—	20	28.0	24.1	23.7	—
81	19.5	16.5	15.2	—	21	28.2	24.3	23.9	—
82	19.7	16.7	15.4	11	22	28.4	24.6	24.1	—
83	20.0	16.9	15.7	—	23	28.6	24.9	24.3	—
84	20.2	17.1	15.9	—	24	28.9	25.2	24.6	—
85	20.4	17.3	16.1	—	25	29.1	25.5	24.8	16
86	20.7	17.4	16.4	—	26	29.3	—	25.0	—
87	20.9	17.6	16.6	—	27	29.5	—	25.2	—
88	21.1	17.8	16.8	—	28	29.7	—	25.4	—
89	21.4	18.0	17.1	—	29	29.9	—	25.6	—
90	21.6	18.2	17.3	12	30	30.1	—	25.8	—

TABLE XI.—*Table to Oechsle's acrometer for must.*

Specific gravity.	Degrees of Oechsle's acrometer for must.	Percentage of crystallized grape-sugar.	Specific gravity.	Degrees of Oechsle's acrometer for must.	Percentage of crystallized grape-sugar.	Specific gravity.	Degrees of Oechsle's acrometer for must.	Percentage of crystallized grape-sugar.	Specific gravity.	Degrees of Oechsle's acrometer for must.	Percentage of crystallized grape-sugar.
1041	41	8.0	1059	59	13.0	1076	76	17.2	1093	93	21.7
1042	42	8.3	1060	60	13.2	1077	77	17.5	1094	94	21.9
1043	43	8.6	1061	61	13.4	1078	78	17.8	1095	95	22.2
1044	44	8.9	1062	62	13.6	1079	79	18.0	1096	96	22.5
1045	45	9.2	1063	63	13.9	1080	80	18.3	1097	97	22.7
1046	46	9.4	1064	64	14.0	1081	81	18.5	1098	98	23.0
1047	47	9.7	1065	65	14.2	1082	82	18.8	1099	99	23.2
1048	48	9.9	1066	66	14.4	1083	83	19.1	1100	100	23.4
1049	49	10.2	1067	67	14.7	1084	84	19.4	1101	101	23.7
1050	50	10.5	1068	68	15.0	1085	85	19.7	1102	102	23.9
1051	51	10.8	1069	69	15.2	1086	86	20.0	1103	103	24.2
1052	52	11.1	1070	70	15.5	1087	87	20.2	1104	104	24.5
1053	53	11.4	1071	71	15.8	1088	88	20.4	1105	105	24.8
1054	54	11.7	1072	72	16.1	1089	89	20.7	1106	106	25.0
1055	55	11.9	1073	73	16.3	1090	90	20.9	1107	107	25.2
1056	56	12.2	1074	74	16.5	1091	91	21.2	1108	108	25.4
1057	57	12.5	1075	75	16.9	1092	92	21.4	1109	109	25.7
1058	58	12.7									

TABLE XII.—*To Massonfour's aerometer.*

Degrees, according to Massonfour.	Weight of a liter, grammes.	Degrees, according to Massonfour.	Weight of a liter, grammes.	Degrees, according to Massonfour.	Weight of a liter, grammes.
1	1008	8	1059	15	1116
2	1015	9	1067	16	1125
3	1022	10	1075	17	1134
4	1029	11	1083	18	1143
5	1036	12	1091	19	1152
6	1043	13	1099	20	1161
7	1051	14	1107		

TABLE XIII.—*For comparing per cent. of sugar with per cent. of extract and the specific gravity. By Pillitz.*

Sugar, per cent. (Pillitz).	Extract, per cent. (Balling).	Specific gravity.	Sugar, per cent. (Pillitz).	Extract, per cent. (Balling).	Specific gravity.	Sugar, per cent. (Pillitz).	Extract, per cent. (Balling).	Specific gravity.
0	4.3	1.0172	9	13.3	1.0543	18	22.3	1.0930.
1	5.3	1.0212	10	14.3	1.0585	19	23.3	1.0975
2	6.3	1.0253	11	15.3	1.0627	20	24.3	1.1017
3	7.3	1.0294	12	16.3	1.0670	21	25.3	1.1060
4	8.3	1.0335	13	17.3	1.0713	22	26.3	1.1103
5	9.3	1.0376	14	18.3	1.0757	23	27.3	1.1146
6	10.3	1.0417	15	19.3	1.0800	24	28.3	1.1189
7	11.3	1.0459	16	20.3	1.0844	25	29.3	1.1232
8	12.3	1.0501	17	21.3	1.0887			

TABLE XIV.—*For determining the content of per cent. of acetic acid contained in a vinegar of — specific gravity.*

*Temperature 15° C. (59° F.).*

(According to A. C. Oudemans.)

Anhydrous acetic acid, per cent.	Specific gravity.	Anhydrous acetic acid, per cent.	Specific gravity.	Anhydrous acetic acid, per cent.	Specific gravity.
100	1.0553	66	1.0717	32	1.0436
99	1.0580	65	1.0712	31	1.0424
98	1.0604	64	1.0707	30	1.0412
97	1.0625	63	1.0702	29	1.0400
96	1.0644	62	1.0697	28	1.0388
95	1.0660	61	1.0691	27	1.0375
94	1.0674	60	1.0685	26	1.0363
93	1.0686	59	1.0679	25	1.0350
92	1.0696	58	1.0673	24	1.0337
91	1.0705	57	1.0666	23	1.0324
90	1.0713	56	1.0660	22	1.0311
89	1.0720	55	1.0653	21	1.0298
88	1.0726	54	1.0646	20	1.0284
87	1.0731	53	1.0638	19	1.0270
86	1.0736	52	1.0631	18	1.0256
85	1.0739	51	1.0623	17	1.0242
84	1.0742	50	1.0615	16	1.0228
83	1.0744	49	1.0607	15	1.0214
82	1.0746	48	1.0598	14	1.0200
81	1.0747	47	1.0589	13	1.0185
80	1.0748	46	1.0580	12	1.0171
79	1.0748	45	1.0571	11	1.0157
78	1.0748	44	1.0562	10	1.0142
77	1.0748	43	1.0552	9	1.0127
76	1.0747	42	1.0543	8	1.0113
75	1.0746	41	1.0533	7	1.0098
74	1.0744	40	1.0523	6	1.0083
73	1.0742	39	1.0513	5	1.0067
72	1.0740	38	1.0502	4	1.0052
71	1.0737	37	1.0492	3	1.0037
70	1.0733	36	1.0481	2	1.0022
69	1.0729	35	1.0470	1	1.0007
68	1.0725	34	1.0459	0	0.9992
67	1.0721	33	1.0447		



TABLE XV.—*For determining the content of per cent. of acetic acid contained in a vinegar of — specific gravity.*

(According to Mohr.)

Anhydrous acetic acid, per cent.	Specific gravity.	Anhydrous acetic acid, per cent.	Specific gravity.	Anhydrous acetic acid, per cent.	Specific gravity.
100	1.0635	66	1.0690	33	1.0440
99	1.0655	65	1.0680	32	1.0420
98	1.0670	64	1.0680	31	1.0410
97	1.0680	63	1.0680	30	1.0400
96	1.0690	62	1.0670	29	1.0390
95	1.0700	61	1.0670	28	1.0380
94	1.0706	60	1.0670	27	1.0360
93	1.0708	59	1.0660	26	1.0350
92	1.0716	58	1.0660	25	1.0340
91	1.0721	57	1.0650	24	1.0330
90	1.0730	56	1.0640	23	1.0320
89	1.0730	55	1.0640	22	1.0310
88	1.0730	54	1.0630	21	1.0290
87	1.0730	53	1.0630	20	1.0270
86	1.0730	52	1.0620	19	1.0260
85	1.0730	51	1.0610	18	1.0250
84	1.0730	50	1.0600	17	1.0240
83	1.0730	49	1.0590	16	1.0230
82	1.0730	48	1.0580	15	1.0220
81	1.0732	47	1.0560	14	1.0200
80	1.0735	46	1.0550	13	1.0180
79	1.0735	45	1.0550	12	1.0170
78	1.0733	44	1.0540	11	1.0160
77	1.0732	43	1.0530	10	1.0150
76	1.0730	42	1.0520	9	1.0130
75	1.0720	41	1.0510	8	1.0120
74	1.0720	40	1.0510	7	1.0100
73	1.0720	39	1.0500	6	1.0080
72	1.0710	38	1.0490	5	1.0070
71	1.0710	37	1.0480	4	1.0050
70	1.0700	36	1.0470	3	1.0040
79	1.0700	35	1.0460	2	1.0020
68	1.0700	34	1.0450	1	1.0010
67	1.0690				

TABLE XVI.—*Comparison of the scales of Réaumur's, Celsius's, and Fahrenheit's thermometers.*

Réaumur.	Celsius.	Fahrenheit.	Réaumur.	Celsius.	Fahrenheit.
—15	—18.75	—	33	41.25	106.25
14	17.50	0.50	34	42.50	108.50
13	16.25	2.75	35	43.75	110.75
12	15.00	5.00	36	45.00	113.00
11	13.75	7.25	37	46.25	115.25
10	12.50	9.50	38	47.50	117.50
9	11.25	11.75	39	48.75	119.75
8	10.00	14.00	40	50.00	122.00
7	8.75	16.25	41	51.25	124.25
6	7.50	18.50	42	52.50	126.50
5	6.25	20.75	43	53.75	128.75
4	5.00	23.00	44	55.00	131.00
3	3.75	25.25	45	56.25	133.25
2	2.50	27.50	46	57.50	135.50
1	1.25	29.75	47	58.75	137.75
0	0	32.00	48	60.00	140.00
+1	+1.25	34.25	49	61.25	142.25
2	2.50	36.50	50	62.50	144.50
3	3.75	38.75	51	63.75	146.75
4	5.00	41.00	52	65.00	149.00
5	6.25	43.25	53	66.25	151.25
6	7.50	45.50	54	67.50	153.50
7	8.75	47.75	55	68.75	155.75
8	10.00	50.00	56	70.00	158.00
9	11.25	52.25	57	71.25	160.25
10	12.50	54.50	58	72.50	162.50
11	13.75	56.75	59	73.75	164.75
12	15.00	59.00	60	75.00	167.00
13	16.25	61.25	61	76.25	169.25
14	17.50	63.50	62	77.50	171.50
15	18.75	65.75	63	78.75	173.75
16	20.00	68.00	64	80.00	176.00
17	21.25	70.25	65	81.25	178.25
18	22.50	72.50	66	82.50	180.50
19	23.75	74.75	67	83.75	182.75
20	25.00	77.00	68	85.00	185.00
21	26.25	79.25	69	86.25	187.25
22	27.50	81.50	70	87.50	189.50
23	28.75	83.75	71	88.75	191.75
24	30.00	86.00	72	90.00	194.00
25	31.25	88.25	73	91.25	196.25
26	32.50	90.50	74	92.50	198.50
27	33.75	92.75	75	93.75	200.75
28	35.00	95.00	76	95.00	203.00
29	36.25	97.25	77	96.25	205.25
30	37.50	99.50	78	97.50	207.50
31	38.75	101.75	79	98.75	209.75
32	40.00	104.00	80	100.00	212.00

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